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EPA

Petroleum Refineries Waste Water Treatment System

Emission Test Report

Golden West Refining Company Sante Fe Springs, California

Contract No. 68-02-3545
Work Assignment 14

EMB Report No. 83 WWS 4

EPA Task Manager

W. E. Kelly

EMISSION TEST REPORT

PETROLEUM REFINERY WASTEWATER TREATMENT SYSTEM

GOLDEN WEST REFINING COMPANY

SANTA FE SPRINGS, CALIFORNIA

Contractor

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GLOSSARY OF TERMS

EMB - Emission Measurement Branch
EPA - Environmental Protection Agency
NSPS - New Source Performance Standard
Golden West - Golden West Refinery Company
IAF - Induced Air Flotation
VOC - Volatile Organic Carbon
COD - Chemical Oxygen Demand
TOC - Total Organic Carbon
TCO - Total Chromatographic Organics
FID - Flame Ionization Detector
GC - Gas Chromatograph
MS - Mass Spectrometer
THC - Total Hydrocarbon
OVA - Organic Vapor Analyzer
TCD - Thermal Conductivity Detection
VOA - Void of Air
QA - Quality Assurance
QC - Quality Control
As C_3H_8 - Based on Propane Standard
ppm - Parts per million
scfm - Standard cubic feet per minute

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1. INTRODUCTION

Under Section 111 of the Clean Air Act, the Environmental Protection Agency is required to develop standards of performance for stationary sources that have been determined to contribute significantly to air pollution. EPA is conducting a study to develop standards that would limit volatile organic compound emissions from new waste water treatment systems in petroleum refineries. Under contract to the Emission Measurement Branch, EPA, TRW Environmental Operations personnel conducted a testing program at the waste water treatment system at Golden West Refinery Company, Santa Fe Springs, California during August 15 to 19, 1983.

The purpose of the test program was to provide estimates of the organic compound release rate from the induced air flotation (IAF) unit. This information is necessary to estimate uncontrolled emission rates from uncovered flotation devices for potential emission reduction and cost effectiveness calculations.

The IAF at Golden West is equipped with a cover with access doors that can be tightly sealed. Plant air is introduced at the influent end of the unit to purge organic vapors from the unit. The ventilation air stream exit the IAF through a vent at the effluent end of the IAF. This vent stream is assisted by a blower which drives the organic laden stream to low pressure nozzles in a nearby fired heater. The ventilation air stream was measured to estimate the organic release rate that would have occurred if the flotation device had been uncovered. This approach was used to estimate uncovered unit emission because of the difficulty in measuring a dispersed fugitive emission. It is assumed that the dominant factors affecting organic emission rates are the water characteristics and the physical turbulence caused by bubbling air through the water, and that meterological factors such as air temperature and wind speed are secondary parameters.

Tests were conducted to determine the mass flow rate and the organic species composition of the ventilation air from the IAF. During these measurements, samples of waste water were collected to characterize the streams from the IAF influent and effluent, and the API separation influent. These samples were analyzed for chemical oxygen demand (COD), total organic carbon (TOC), total chromatographical organics (TCO), and oil and grease content using standard methods for water analysis.

The results of these tests are presented in Section 2. A description of the process and the operation during the test period is given in Section 3. The sampling locations and the sampling and analytical procedures are discussed in Sections 4 and 5 respectively. The appendices to this report contain example calculations, field data, test logs and a list of project participants.

2. SUMMARY AND DISCUSSION OF RESULTS

This section details the results of the testing and analysis at Golden West Refinery Company waste water treatment unit. The refinery waste water treatment system is illustrated in Figure 3-1, and the sampling locations are indicated in Figure 2-1. Table 2-1 presents a summary of the periods during which continuous hydrocarbon monitoring was performed, and the periods during which integrated gas samples were collected, flow rate measurements were performed, and when liquid samples were collected. The results are presented in separate sections for air and water samples.

2.1 VENTILATION AIR SAMPLING RESULTS

A summary of the daily average total hydrocarbon mass flow rates in the IAF ventilation air stream is presented in Table 2-2. The total hydrocarbon measurement does not exclude methane. The hydrocarbon mass flow in the IAF ventilation air ranged from 1.17 to 1.40 lbs/hr (24 hr. basis) and averaged 1.31 lbs/hr over the five days of testing. The test results on a one-hour average basis are presented in Tables 2-3 to 2-7. The average total hydrocarbon concentration based on equivalents of propane is presented for each one-hour period. Propane was chosen as the calibration species because it is a stable compound and calibration mixtures are easily acquired and stored. For the organic species expected at refineries, the response of the analysis is directly proportional to the carbon content. While the concentration results are on a propane basis and are not equal to the time hydrocarbon concentration, the calculated mass flow rates are equivalent to true hydrocarbon mass flow rates.

The average gaseous flow rate result that was used for calculation of the hydrocarbon mass flow is also given for each day of monitoring. This value represents the average flow over a two to six hour test

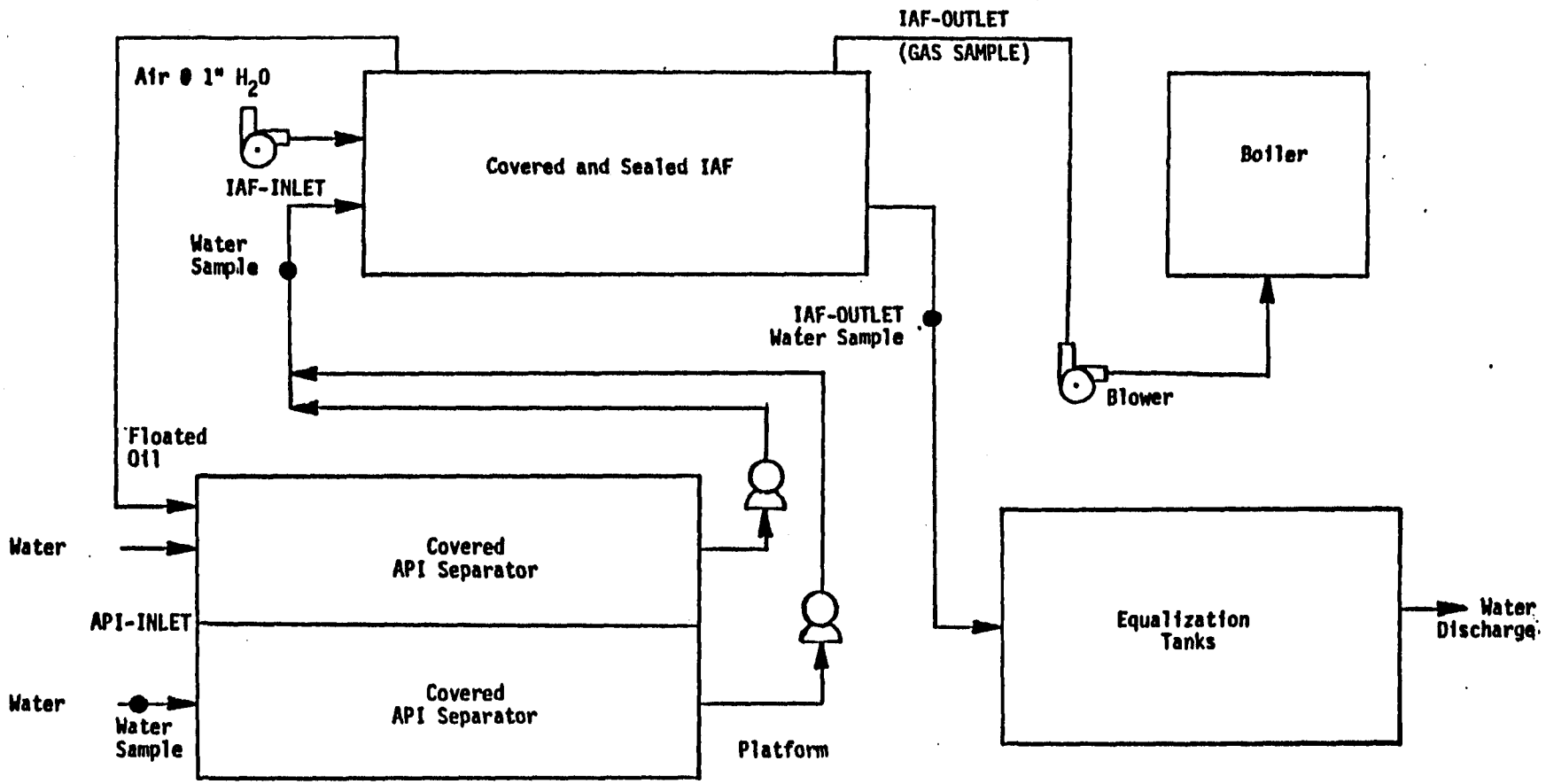
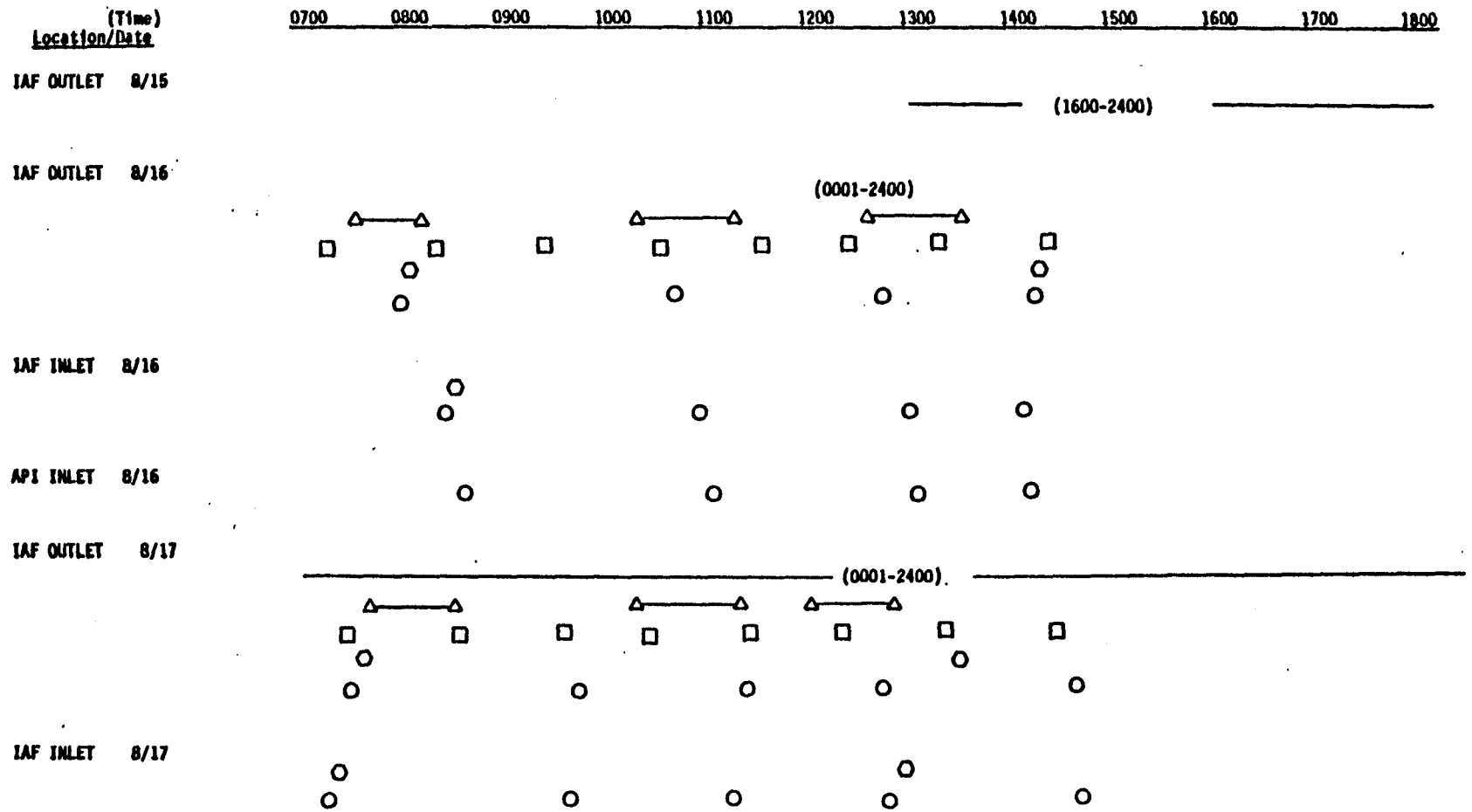


Figure 2-1. Wastewater treatment facilities with sample locations at Santa Fe Springs, California.

Table 2-1. DAILY TIME TABLE OF SAMPLING ACTIVITIES AT GOLDEN WEST - SANTA FE SPRINGS, CALIFORNIA



LEGEND

- △ (Method 18-Gas Bag)
- □ (Velocity-anemometer)
- O (Liquid Composite)
- O (Liquid VOA)
- ( ) (Method 25-A THC)

Table 2-1. Concluded

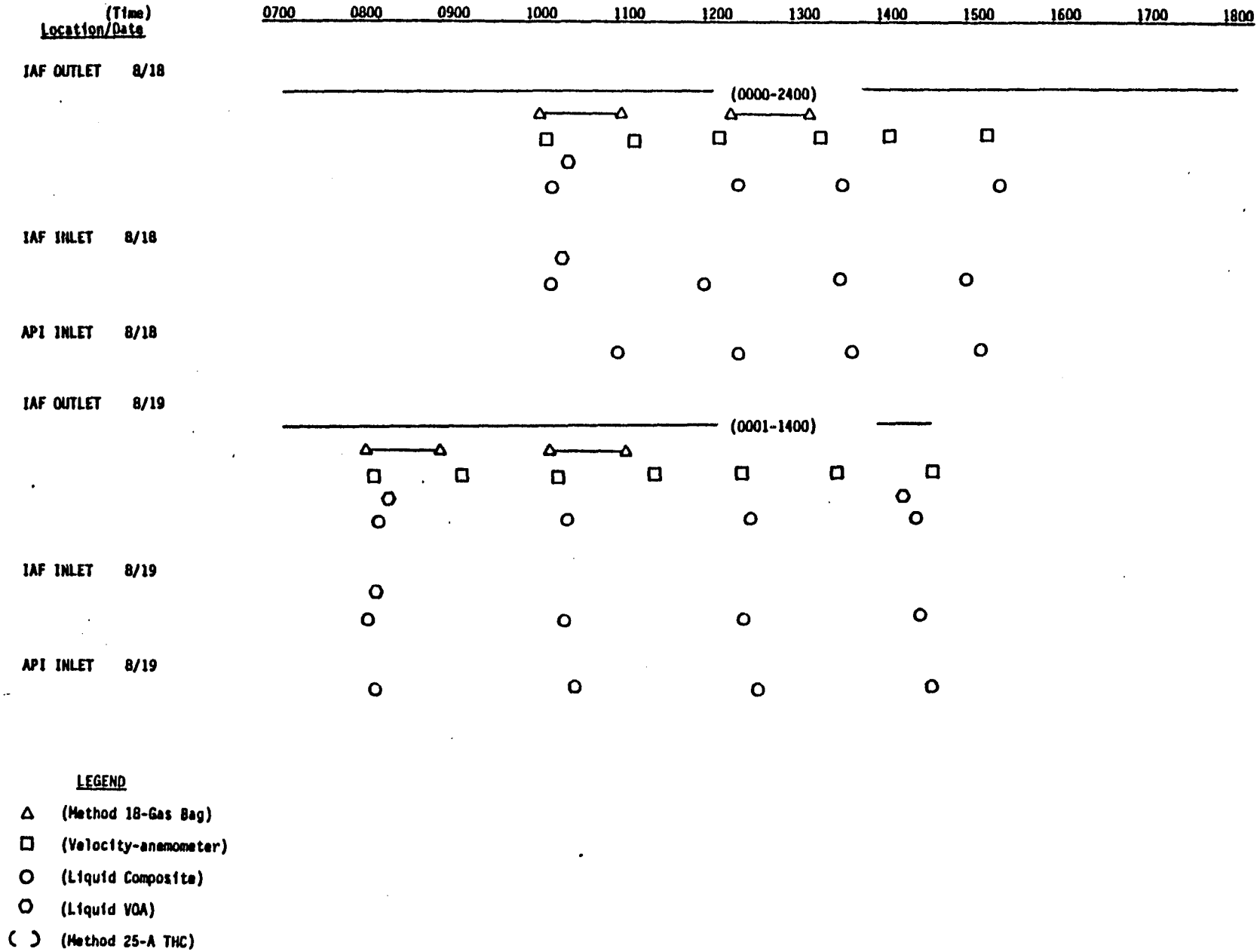


Table 2-2. DAILY EMISSION RATE AVERAGES AT THE IAF OUTLET SAMPLE LOCATION ON TEST DAYS 8/15/83 to 8/19/83 GOLDEN WEST REFINERY - SANTA FE SPRINGS, CALIFORNIA

Test day	Average daily emission rate (lb/hr as C_3H_8)
8/15/83	1.40
8/16/83	1.39
8/17/83	1.17
8/18/83	1.25
8/19/83	1.39

Table 2-3. CONTINUOUS EMISSION RESULTS: HYDROCARBON MONITORING AT THE IAF OUTLET SAMPLE LOCATION - GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA - TEST DAY 8/15/83

Time	Concentration[a] (ppm as C_3H_8)	Flow[b] (SCFM)	Emission rate (lbs/hr as C_3H_8)
1600[c]	7092	30.4	1.38
1700	7042	30.4	1.53
1800	6925	30.4	1.50
1900	6792	30.4	1.47
2000	6400	30.4	1.39
2100	6258	30.4	1.36
2200	6170	30.4	1.34
2300	6083	30.4	1.32
2400	6050	30.4	1.31
Average			1.40

[a]Concentration is average value for continuous readings across the hour (0-55 minutes) based on 5-minute readings.

[b]No flow measurement on 8/15/83; therefore, used average of IAF daily flows. See Table 2-8.

[c]Continuous Hydrocarbon Analyzer (Beckman 402) on-line at IAF outlet starting the test period.

Table 2-4. CONTINUOUS EMISSION RESULTS: HYDROCARBON MONITORING AT THE IAF OUTLET SAMPLE LOCATION - GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA - TEST DAY 8/16/83

Time	Concentration[a] (ppm as C_3H_8)	Flow[b] (SCFM)	Emission rate (lbs/hr as C_3H_8)
0100	6250	33.9	1.36
0200	6125	33.9	1.33
0300	6100	33.9	1.32
0400	6300	33.9	1.37
0500	6750	33.9	1.47
0600	6675	33.9	1.45
0700	6844	33.9	1.49
0800	6700	33.9	1.45
0900	6458	33.9	1.40
1000	6725	33.9	1.46
1100	7483	33.9	1.62
1200	7267	33.9	1.58
1300	6908	33.9	1.50
1400	6289	33.9	1.37
1500	5850	33.9	1.27
1600	5850	33.9	1.27
1700	6058	33.9	1.31
1800	5725	33.9	1.24
1900	5650	33.9	1.23
2000	6100	33.9	1.32
2100	6233	33.9	1.35
2200	6300	33.9	1.37
2300	6708	33.9	1.46
2400	6883	33.9	1.49
Average			1.39

[a]Concentration is average value for continuous readings across the hour (0-55 minutes) based on 5-minute readings.

[b]Two flow measurements taken on 8/16/83; therefore, average used for daily flow. See Table 2-8.

Table 2-5. CONTINUOUS EMISSION RESULTS: HYDROCARBON MONITORING AT THE IAF OUTLET SAMPLE LOCATION - GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA - TEST DAY 8/17/83

Time	Concentration[a] (ppm as C_3H_8)	Flow[b] (SCFM)	Emission rate (lbs/hr as C_3H_8)
0100	6925	25.7	1.14
0200	6783	25.7	1.12
0300	6075	25.7	1.01
0400	6608	25.7	1.09
0500	6950	25.7	1.14
0600	7033	25.7	1.16
0700	7150	25.7	1.17
0800	6867	25.7	1.13
0900	7992	25.7	1.32
1000	8675	25.7	1.43
1100	8592	25.7	1.41
1200	8811	25.7	1.45
1300	7500	25.7	1.23
1400	6508	25.7	1.07
1500	6558	25.7	1.08
1600	6175	25.7	1.02
1700	6367	25.7	1.04
1800	6667	25.7	1.10
1900	6958	25.7	1.14
2000	7308	25.7	1.20
2100	7392	25.7	1.21
2200	7017	25.7	1.15
2300	6825	25.7	1.12
2400	6917	25.7	1.14
Average			1.17

[a]Concentration is average value for continuous readings across the hour (0-55 minutes) based on 5-minute readings.

[b]One flow measurement taken across day period (7:40-14:19) on 8/17/83 and used as average daily flow. See Table 2-8.

Table 2-6. CONTINUOUS EMISSION RESULTS: HYDROCARBON MONITORING AT THE IAF OUTLET SAMPLE LOCATION - GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA - TEST DAY 8/18/83

Time	Concentration[a] (ppm as C_3H_8)	Flow[b] (SCFM)	Emission rate (lbs/hr as C_3H_8)
0100	7192	28.7	1.32
0200	7300	28.7	1.34
0300	7483	28.7	1.37
0400	7500	28.7	1.37
0500	7473	28.7	1.32
0600	6942	28.7	1.27
0700	6800	28.7	1.25
0800	c	—	—
0900	c	—	—
1000	6010	28.7	1.11
1100	5940	28.7	1.09
1200	6475	28.7	1.19
1300	6650	28.7	1.22
1400	6800	28.7	1.25
1500	7150	28.7	1.31
1600	7050	28.7	1.29
1700	6750	28.7	1.24
1800	6700	28.7	1.25
1900	6440	28.7	1.18
2000	6710	28.7	1.23
2100	6650	28.7	1.22
2200	6630	28.7	1.22
2300	6750	28.7	1.24
2400	6660	28.7	1.22
Average			1.25

[a]Concentration is average value for continuous readings across the hour (0-55 minutes) based on 5-minute readings.

[b]One flow measurement taken across day period (10:25-15:03) on 8/18/83 and used as average daily flow. See Table 2-8.

[c]Process interruption with instruments off-line.

Table 2-7. CONTINUOUS EMISSION RESULTS: HYDROCARBON MONITORING AT THE IAF OUTLET SAMPLE LOCATION - GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA - TEST DAY 8/19/83

Time	Concentration[a] (ppm as C_3H_8)	Flow[b] (SCFM)	Emission rate (lbs/hr as C_3H_8)
0100	6440	34.5	1.42
0200	6210	34.5	1.37
0300	5920	34.5	1.31
0400	5900	34.5	1.30
0500	6090	34.5	1.35
0600	6150	34.5	1.36
0700	6360	34.5	1.41
0800	6200	34.5	1.37
0900	6210	34.5	1.37
1000	6325	34.5	1.40
1100	6630	34.5	1.46
1200	6760	34.5	1.49
1300[c]	6640	34.5	1.47
Average			1.39

[a]Concentration is average value for continuous readings across the hour (0-55 minutes) based on 5-minute readings.

[b]One flow measurement taken across day period (8:34-14:03) on 8/19/83 and used as average daily flow. See Table 2-8.

[e]End of test at the IAF Outlet/Golden West Refinery.

period each day of testing. The results of the flow measurement are presented in Table 2-8. A single average value can be used because the flow rate was determined to be constant based on intermediate readings during the test. The field data sheets containing intermediate measurements are contained in Appendix A.

The results of the analysis of the integrated gas samples of the IAF ventilation air are presented in Tables 2-9 and 2-10. The species analyses were obtained using two field gas chromatographic systems and were intended to generally identify the major components and their approximate concentrations. Calibration standards were available for C1 to C5, benzene and m-xylene, so the results for these compounds can be calculated directly. Hexane, heptane, and p-xylene are calculated as equivalents of the nearest carbon number calibration species. Other peaks were also grouped with the closest eluting calibration species for computation. Since a benzene standard was used to establish a specific retention time for that compound, it can be concluded that the peak occurring at that time was benzene. However, these are some compounds found at refineries that tend to elute near benzene (such as methylcyclopentane and cyclohexane) and would be indistinguishable with this analytical system. However, since clear identification of toluene and xylene were present, it is probable that at least part of the concentrations attributed to benzene was actually benzenes. Additional descriptions of the chromatographic techniques are given in Section 5.

The general results of the species analysis are consistent in the relative amounts of the compounds found. The major peaks were distributed in the C4 to C7 range. The results of these analyses can be used to calculate a non-methane hydrocarbon emission rate, but this calculation was not performed for this report.

The results of chromatographic analysis to determine the balance gas composition are generally as expected, with the analysis being approximately air. However, several of the analysis results show oxygen concentrations greater than found in ambient air. These higher results are probably due to a variation in the operation conditions of the analyzer between calibrations and do not represent real values. This has no affect on any of the other calculated results.

Table 2-8. FLOW MEASUREMENTS WITH ANEMOMETER:
GOLDEN WEST - SANTA FE SPRINGS, CALIFORNIA

Date	Time	Temperature (°F)	Feet	Time period in min	Anemometer average rate (ft/min)		Actual volumetric flowrate (ACFM)	Standard volumetric flowrate (SCFM)
					Indicated	True		
8/16/83	0800-1200	123	110300	265	416	416	36.3	32.7
8/16/83	1200-1400	128	46900	104	450	450	39.3	35.1
8/17/83	0740-1419	117	125500	399	314	324	28.3	25.7
8/18/83	1026-1503	116	98000	277	354	360	31.4	28.7
8/19/83	0834-1403	115	142000	329	432	432	37.7	34.5

Table 2-9. GAS CHROMATOGRAPH RESULTS FROM THE IAF TEST DAYS - 8/16/83 AND 8/17/83 GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA

DATE	8/16	8/16	8/16	8/17	8/17[a]	8/17[a]
TIME	735-835	1020-1120	1235-1335	0745-0845	1000-1100	1153-1253
RUN NO.	1	2	3	1	2	3
ANALYTICAL RESULTS (ppmv as compound)						
C-1	74.0	110	90.8	138	135	262
C-2	6.8	9.4	9.6	7.8	20.9	
C-3	14.2	22.1	14.4	19.0	78.5	122
C-4	38.6	269	108	140	315	365
C-5	52.0	250	130	183	685	341
Hexane	115	370	1068	180	577	524
Benzene	1357	2851	2424	1758	3638	3530
Toluene	1346	2486	2321	1629	2376	2476
m-Xylene	933	1458	1578	905	813	885
o-Xylene	326	467	510	305	283	308
TOTAL HYDROCARBON[b] (ppmv as compound)	4262	8292	8253	5265	8921	8813
CONTINUOUS MONITOR DATA						
Hydrocarbon Level (ppmv as C_3H_8)	6772	7104	7087	7008	8675	8811
Emission Rate (lb/hr)	1.47	1.54	1.54	1.15	1.42	1.45
PROCESS CONDITIONS						
% N_2	76.17	75.69	73.46	75.92	73.75	73.09
% O_2	18.60	19.50	18.52	21.66	20.04	21.12
% CO_2			0.94			0.40

[a]See explanation in Section 2.2.2.

[b]Total includes unidentified hydrocarbon responsive to GC/FID.

Table 2-10. GAS CHROMATOGRAPH RESULTS FROM THE IAF
TEST DAYS - 8/18/83 AND 8/19/83
GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA

DATE	8/18	8/18	8/19	8/19
TIME	1030-1146	1310-1410	850-950	1030-1130
RUN NO.	1	2	1	2
ANALYTICAL RESULTS (ppmv as compound)				
C-1	44.5	94.7	66.0	72.8
C-2	3.0	4.1	5.3	6.8
C-3	4.2	8.0	8.1	10.0
C-4	10.5	96.5	28.4	50.7
C-5	14.9	71.0	90.3	78.9
Hexane	49.7	81.4	93.5	116
Benzene	547	1106	865	1236
Toluene	889	1661	1110	1785
m-Xylene	647	1164	640	890
o-Xylene	236	407	228	297
TOTAL HYDROCARBON[a] (ppmv as compound)	2446	4695	3135	4544
CONTINUOUS MONITOR DATA				
Hydrocarbon Level (ppmv as C_3H_8)	5975	6725	6205	6327
Emission Rate (lb/hr)	1.08	1.21	1.37	1.43
PROCESS CONDITIONS				
% N_2	75.75	72.69	73.61	70.92
% O_2	21.57	21.21	23.43	27.74
% CO_2	0.76	1.18	0.43	0.33

[a]Total includes unidentified hydrocarbon responsive to GC/FID.

2.2 PROCESS WATER ANALYSES

Tables 2-11 through 2-15 provide the process water analysis for the composite and grab samples taken during the hydrocarbon (air) monitoring.

Designated samples (item, location) were analyzed for the following parameters:

- TOC (total organic carbon);
- COD (chemical oxygen demand);
- oil and grease; and
- TCO (total chromatographable organics/hydrocarbon speciation (C_7-C_{30}) and VOA by purge and trap GC/FID.

All analytical parameters are reported in milligrams per liter (ppmw), except purge and trap values which are given in parts per billion (ppbw).

The most critical factors in the measurement of the process water parameters were the collection of representative samples at the site location and obtaining a representative aliquot for analysis in the laboratory. In most cases the samples involved two-phase oil/water mixtures which contributed to the non-homogeneity of the samples and to the variation in the sample values.

The sampling points at the Golden West treatment plant were dictated by the physical layout and available sample locations. The samples were collected from streams at elevated temperatures, stored on ice, and shipped to the TRW laboratory. Sample preservatives were not utilized in preference to immediate analysis (24-48 hours) and to the elevated levels of hydrocarbons in the streams. Upon arrival at the laboratory all samples were homogenized prior to analysis; however, the two-phased system and the cooling of the sampling affected the homogeneity of the samples. All samples were brought to room temperature and shaken vigorously before samples were removed. In addition, due to the high levels of the parameters being measured, the size of the sample aliquots were small which also contributed to the variability from sample to sample.

Table 2-11. GOLDEN WEST SAMPLES TAKEN ON 8/16/83

	TRW No.	COD mg/L	Oil/grease mg/L	TOC mg/L	TCO mg/L
Liquid Composite Samples					
IAF-in	5,047	2,323	11.31	—	104.46
IAF-out	5,051	909	21.89	—	40.78
API-in	5,043	2,020	23.37	—	25.64
Volatile Organic Samples					
IAF-in VOA (0805)	5,055	—	—	344	—
IAF-in VOA (1400)	5,056	—	—	411	—
IAF-out VOA (0805)	5,063	—	—	237	—
IAF-out VOA (1400)	5,064	—	—	304	—

(continued)

Table 2-11. Concluded

	TRW No.		mg/L
Liquid Composite Samples			
IAF-in	5,047	Toluene	7.611
		C_8	5.581
		C_{10}	28.782
		C_{10}	8.904
		C_{10}	6.967
		C_{12}	11.572
		C_{13}	12.999
		C_{13}	3.990
		C_{14}	6.041
		C_{15}	11.920
		C_{16}	5.032
		C_{17}	229.816
		C_{18}	60.938
		C_{19}	65.569
		C_{20}	34.653
		C_{21}	34.247
		C_{22}	24.253
IAF-out	5,051	Toluene	3.721
		C_8	1.841
		C_9	0.899
		C_9	21.115
		C_{10}	6.998
		C_{10}	13.501
		C_{12}	1.888
API-in	5,043	Toluene	2.546
		C_8	1.632
		C_9	5.749
		C_{10}	3.522
		C_{10}	4.173
		C_{12}	2.765
		C_{13}	2.646
		C_{14}	1.699
		C_{15}	2.621
		C_{16}	1.395
		C_{17}	65.244

Table 2-12. GOLDEN WEST SAMPLES TAKEN ON 8/17/83

	TRW No.	COD mg/L	Oil/grease mg/L	TOC mg/L	TCO mg/L
Liquid Composite Samples					
IAF-in	5,048	4,089	14.09	—	158.5
IAF-out	5,052	2,328	4.59	—	109.32
API-in	5,044	5,628	17.62	—	244.30
Volatile Organic Samples					
IAF-in VOA (0740)	5,057	—	—	554	—
IAF-in VOA (1300)	5,058	—	—	426	—
IAF-out VOA (1300)	5,065	—	—	323	—
IAF-out VOA (0740)	5,066	—	—	137	—

(continued)

Table 2-12. Continued

	TRW No.		mg/L
Liquid Composite Samples			
IAF-in	5,048	Toluene	76.223
		C_7	1.835
		C_8	3.602
		C_8	2.422
		C_8	2.066
		C_8	5.420
		C_8	17.959
		C_8	6.712
		C_9	3.833
		C_9	1.632
		C_9	2.160
		C_9	2.644
		C_9	3.057
		C_9	4.577
		C_9	2.640
		C_9	5.201
		C_9	5.709
		C_{10}	3.968
		C_{10}	8.078
		C_{10}	11.172
		C_{11}	4.848
		C_{11}	2.108
		C_{11}	3.772
		C_{11}	1.906
		C_{11}	1.556
		C_{11}	2.039
		C_{12}	7.783
		C_{12}	2.979
		C_{12}	2.162
		C_{12}	2.496
		C_{12}	13.111
		C_{12}	14.532
		C_{13}	7.058
		C_{13}	3.105
		C_{14}	4.510
		C_{14}	3.376
		C_{15}	10.791
		C_{15}	4.026
		C_{16}	5.481
		C_{16}	2.347
		C_{17}	91.409
		C_{17}	224.621

(continued)

Table 2-12. Continued

	TRW No.		mg/L
	5,048	C_{18}	87.140
		C_{18}	84.054
		C_{19}	110.444
		C_{20}	73.046
		C_{21}	90.032
		C_{22}	73.718
		C_{23}	46.656
		C_{24}	55.906
		C_{25}	30.594
IAF-out	5,052	Toluene	50.025
		C_7	0.482
		C_7	0.516
		C_7	0.957
		C_8	0.688
		C_8	0.563
		C_8	2.543
		C_8	10.277
		C_8	3.919
		C_8	1.296
		C_9	0.628
		C_9	0.618
		C_9	1.126
		C_9	1.611
		C_9	2.743
		C_9	1.290
		C_9	30.117
		C_{10}	2.226
		C_{10}	2.117
		C_{10}	0.971
		C_{10}	0.588
		C_{10}	0.889
		C_{10}	9.658
		C_{10}	20.001
		C_{10}	2.108
		C_{11}	0.666
		C_{11}	1.663
		C_{11}	2.282
		C_{11}	0.674
		C_{11}	2.144
		C_{11}	0.726
		C_{11}	0.916
		C_{11}	0.681
		C_{11}	1.092
		C_{11}	2.921

(continued)

Table 2-12. Continued

	TRW No.		mg/L
	5,052	C_{12}	1.337
		C_{12}	1.231
		C_{12}	1.445
		C_{12}	7.804
		C_{13}	8.226
		C_{13}	1.390
		C_{13}	1.850
		C_{14}	2.598
		C_{14}	1.808
		C_{15}	5.846
		C_{15}	2.174
		C_{16}	84.094
		C_{17}	105.381
		C_{18}	39.690
		C_{19}	50.973
		C_{20}	36.077
		C_{21}	29.241
		C_{22}	20.598
		C_{23}	23.798
		C_{24}	14.621
API-in	5,044	Toluene	23.873
		C_7	1.593
		C_7	2.085
		C_8	2.157
		C_8	5.764
		C_8	24.131
		C_8	9.263
		C_8	2.470
		C_9	3.303
		C_9	4.726
		C_9	6.821
		C_9	3.696
		C_9	1.205
		C_9	4.956
		C_9	9.215
		C_{10}	5.188
		C_{10}	2.297
		C_{10}	2.867
		C_{10}	1.772
		C_{10}	8.807
		C_{10}	4.265
		C_{10}	2.081

(continued)

Table 2-12. Concluded

TRW No.		mg/L
5,044	C_{11}	3.670
	C_{11}	1.726
	C_{11}	3.837
	C_{11}	4.716
	C_{11}	1.931
	C_{11}	1.812
	C_{11}	5.883
	C_{11}	2.842
	C_{11}	6.898
	C_{12}	2.667
	C_{12}	3.212
	C_{12}	3.528
	C_{12}	2.250
	C_{12}	15.183
	C_{12}	15.331
	C_{13}	7.276
	C_{14}	15.577
	C_{14}	7.765
	C_{15}	3.512
	C_{16}	63.229
	C_{17}	180.452
	C_{18}	86.216

Table 2-13. GOLDEN WEST SAMPLES TAKEN ON 8/18/83

	TRW No.	COD mg/L	Oil/grease mg/L	TOC mg/L	TCO mg/L
Liquid Composite Samples					
IAF-in	5,049	1,162	31.83	—	46.48
IAF-out	5,053	1,111	16.71	—	34.34
API-4 (1130)	5,045	1,364	15.16	—	36.04
Volatile Organic Samples					
IAF-in VOA (1050)	5,059	—	—	204	—
IAF-in VOA (1500)	5,060	—	—	283	—
IAF-out VOA (1050)	5,062[a]	—	—	—	—
IAF-out VOA (1500)	5,067	—	—	315	—

[a]Sample broken in laboratory.

(continued)

Table 2-13. Concluded

	TRW No.		mg/L
Liquid Composite Samples			
IAF-in	5,049	Toluene	9.752
		C_8	4.435
		C_8	1.832
		C_9	1.299
		C_9	22.145
		C_{10}	7.012
		C_{10}	14.987
		C_{12}	2.081
		C_{15}	1.203
		C_{18}	29.697
IAF-out	5,053	Toluene	5.949
		C_8	2.174
		C_9	1.071
		C_9	16,975
		C_{10}	5.575
		C_{10}	10.822
		C_{12}	0.853
API-4 (1130)	5,045	Toluene	5.477
		C_8	2.531
		C_8	0.971
		C_9	1.052
		C_9	17.101
		C_{10}	5.889
		C_{10}	12.505
		C_{12}	1.399
		C_{15}	0.976
		C_{18}	25.959

Table 2-14. GOLDEN WEST SAMPLES TAKEN ON 8/19/83

	TRW No.	COD mg/L	Oil/grease mg/L	TOC mg/L	TCO mg/L
Liquid Composite Samples					
IAF-in	5,050	1,194	348	—	—
IAF-out	5,054	830	332	—	—
		960	201	—	—
API-in	5,046	3,482	1,321	—	—
Volatile Organic Samples					
IAF-in VOA (0830)	5,061	—	—	289	—
IAF-in VOA (1400)	5,070	—	—	509	—
IAF-out VOA (0830)	5,068	—	—	293	—
IAF-out VOA (140)	5,069	—	—	607	—

Table 2-15. C_1 TO C_7 SPECIATION BY GC/FID PURGE AND TRAP
GOLDEN WEST, SANTA FE SPRINGS, CALIFORNIA

TRW No.	Sample Number	Compound	Date Taken	Concentration (in ppb)
5056	IAF-INLET-VOA-1400	C_3H_6O	8/16/83	342
		C_5H_{12}		87.0
		$C_6H_{14}O$		12.6
		Benzene		136
		Hexane		36.8
		C_7H_{14}		18.4
		C_7H_{16}		14.4
		Toluene		243
5064	IAF-OUT-VOA-1400	C_3H_6O	8/16/83	310
		C_5H_{12}		108
		$C_6H_{14}O$		71.2
		Benzene		5710
		Hexane		8.21
		C_7H_{14}		48.8
		C_7H_{16}		166
		Toluene		7620
5057	IAF-INLET-VOA-0740	C_3H_6O	8/17/83	379
		C_5H_{12}		103
		$C_6H_{14}O$		91.0
		Benzene		6450
		Hexane		13.7
		C_7H_{14}		74.0
		C_7H_{16}		11.9
		Toluene		8640

(continued)

Table 2-15. .Concluded

TRW No.	Sample Number	Compound	Date Taken	Concentration (in ppb)
5066	IAF-OUT-VOA-0740	C_3H_6O	8/17/83	359
		C_5H_{12}		106
		$C_6H_{14}O$		73.8
		Benzene		5990
		Hexane		22.5
		C_7H_{14}		80.6
		C_7H_{16}		4.32
		Toluene		8350
5059	IAF-INLET-VOA-1050	C_3H_6O	8/18/83	260
		C_5H_{12}		104
		$C_6H_{14}O$		147
		Benzene		6230
		Hexane		25.2
		C_7H_{14}		136
		C_7H_{16}		15.0
		Toluene		11000
5067	IAF-OUT-VOA-1050	C_3H_6O	8/18/83	234
		C_5H_{12}		102
		Benzene		487
		Toluene		646

3. PROCESS DESCRIPTION

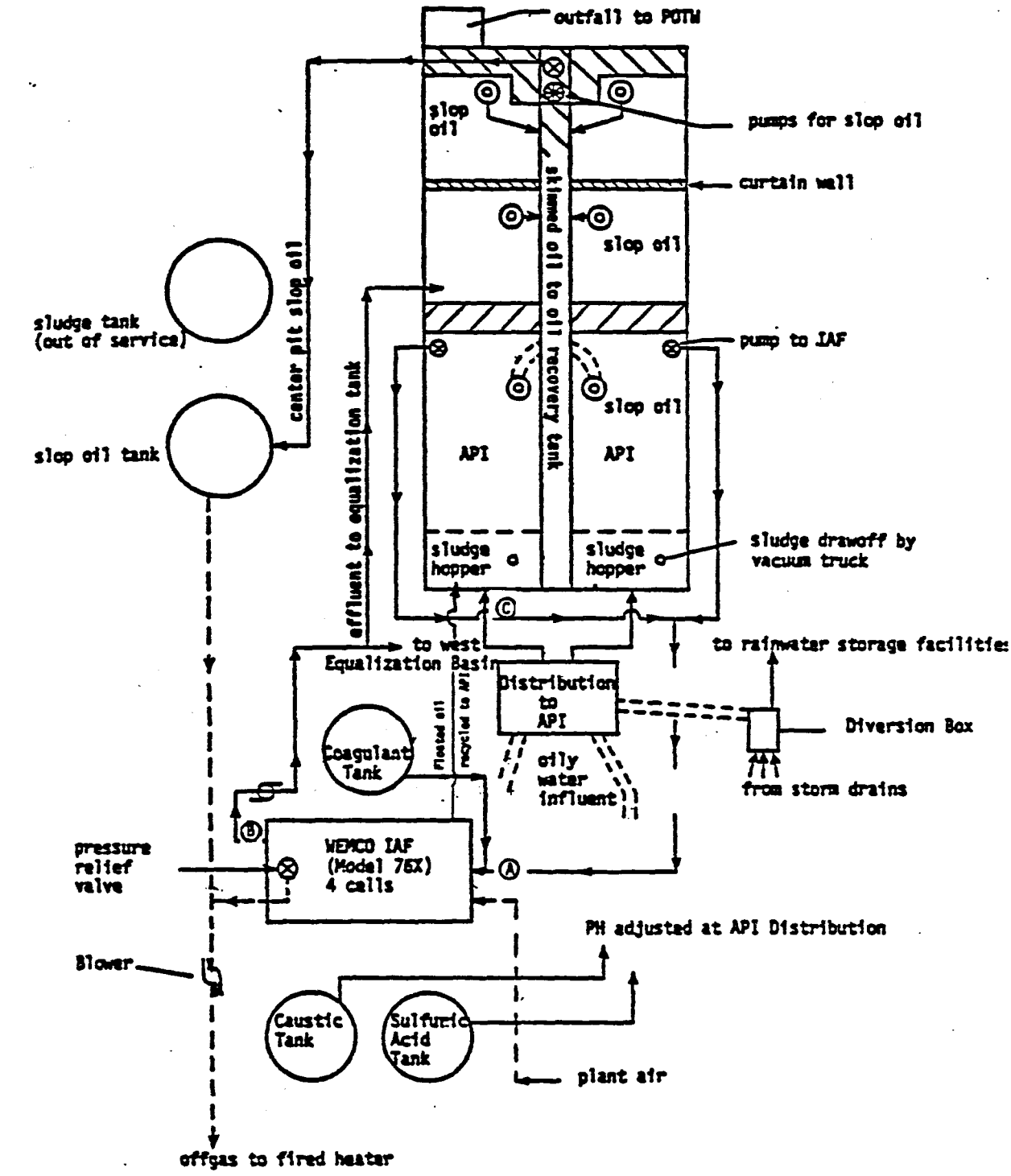
The Golden West Refining Company's Santa Fe Springs facility is a small refinery located in a semi-industrial area of Santa Fe Springs, California. The refinery has a crude throughput capacity of 51,000 barrels per calendar day (b/cd) and was operating at approximately 65 percent capacity during the time of the test. Crude throughputs recorded during the test period are listed in Table 3-1.

The Effluent Guidelines Division of the Environmental Protection Agency places Golden West in refinery subcategory B which includes refineries producing petroleum products by the use of topping and cracking operations. Wastewater produced by the refinery undergoes primary and secondary oil removal before being sent to a Publicly Owned Treatment Works (POTW).

3.1 REFINERY WASTEWATER SYSTEM

The refinery wastewater system at Golden West is shown schematically in Figure 3-1. Oily wastewaters are collected in the process units and directed to the distribution box of the API separators through two trunklines. Stormwaters are segregated from the oily wastewaters as much as possible and are collected in three trunklines which converge on a diversion box located near the API separators. During periods of heavy rainfall, stormwater will be diverted to storage facilities located away from the treatment system. Otherwise, the small quantities of wastewater collected by three storm mains will enter the API distribution box. Sour wastewaters undergo inplant treatment before reaching the API separators. The sour water treatment system is shown in Figure 3-2.

The distribution box divides wastewater flow equally between the two API separators. Each separator is approximately 15 feet wide, 60 feet long, 10 feet deep, and is provided with a wooden cover. Chain



Ⓐ IAF INLET
Ⓑ IAF OUTLET
Ⓒ API INLET

Figure 3-1. Wastewater treatment facility, Golden West Refining Company.

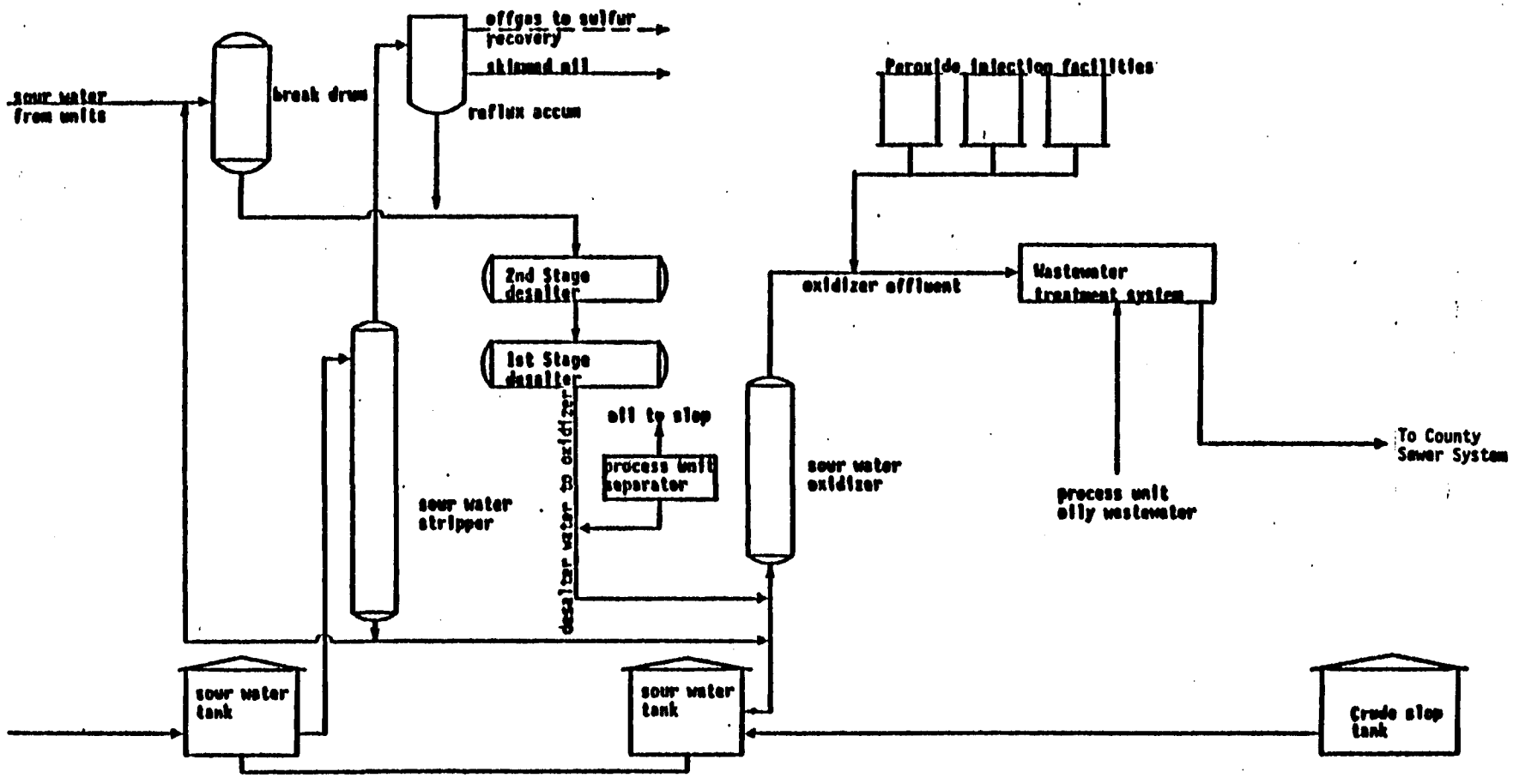


Figure 3-2. Sour water treating system.

Table 3-1. CRUDE THROUGHPUTS DURING THE TEST PERIOD

Date	Barrels per calendar day
8-15-83	34,288
8-16-83	33,939
8-17-83	33,323
8-18-83	33,062
8-19-83	33,233

driven wooden skimming bars continually push floating oil to slop oil pits and scrape settled sludge into sludge hoppers. Oil collected in the slop oil pits flows to a channel located between the separators. Settled sludge is removed from the sludge hoppers by vacuum trucks two to three times per week.

Effluent from the API separators is pumped to the IAF for secondary oil removal. The induced air flotation unit (IAF) was installed by WEMCO and is designed to treat a wastewater flow of 750 gallons per minute (gpm). Small quantities of chemical coagulant are added to the wastewater prior to the IAF. The coagulant aids in breaking oily emulsions and improves the performance of the system. Floated oil removed from the IAF is recirculated to the API separators. Wastewater effluent is pumped to dual open bays which are adjacent to the API separators. The open bays are similar in configuration to the API separators. The bays are uncovered and do not have mechanical skimmers.

The open bays are the final treatment step at Golden West. Each tank is divided into two sections by a curtain wall which acts to retain any floating oil. Slop oil pits located in each section collect the floating oil which then flows into the collection channel between the two bays. All the oil collected in the channel is pumped to a slop oil holding tank located next to the wastewater treatment facilities.

3.2 EMISSION CONTROL TECHNIQUES

Volatile organic compound (VOC) emissions are controlled from the API separators and the IAF system. The separators are equipped with wooden covers and are sealed as tightly as possible. Access doors are used to inspect the operation of the separators. The covers can be easily removed to allow maintenance procedures to be carried out. During the test period, one API separator was emptied and uncovered in order to repair the mechanical skimmer.

The IAF is designed with a cover and eight access doors allow visual inspection of the unit. These doors are gasketed and can be tightly sealed. Plant air is introduced at the influent end of the IAF to purge VOC from the vapor space. The purged vapors leave the IAF through a vent at the effluent end of the unit. This vent leads to a

blower which drives the VOC emissions to the fuel gas input of a fired heater located near the wastewater treatment system. The vent from the IAF and blower are also shown in Figure 3-1.

3.3 MONITORING OF WASTEWATER TREATMENT SYSTEM

The effluent from the wastewater treatment system at Golden West is sent to a Los Angeles County Treatment Works before final disposal. The refinery wastewater system must meet pretreatment requirements specified by the county. Golden West routinely monitors refinery effluent for pH, thiosulfate sulfur, BOD, suspended solids, fluorides, and ammonia. An outside contractor conducts further wastewater analysis on a quarterly basis to insure compliance with the county requirements.

A circular chart records wastewater flow to the IAF. Table 3-2 is a summary of flow readings taken during the test period. Average wastewater flow to the IAF was 450 to 500 gallons per minute. Golden West reported an average flow of 400 to 450 gpm as being the normal range. The slightly higher average flow recorded during the test may be a result of rainfall during the week. The wastewater system in general is capable of handling peak flows of 1000 gpm. This would represent extreme conditions, however, since the IAF is designed to handle only 750 gpm.

Oily wastewater from all process units is directed to the API separators. However, it is impossible to monitor flow from specific units and it is therefore difficult to anticipate upset conditions which would influence the wastewater system. The operators responsible for the system must regularly observe the operation at the API separators, IAF, and open bays to detect upsets. During the test period, one instance of upset condition was observed.

On Wednesday morning, August 17, large amounts of floating oil were observed in the open bays. Normally, very little, if any, floating oil would be seen at this stage of treatment. Investigations by plant operators revealed that an excessive quantity of oil had accidently entered the sour water stripping system. The oil which entered the system during the upset had apparently come from the crude slop tank. Unusually high quantities of oil were being drawn from the tank with the sour waters. Normal procedures at Golden West call for the sour water

Table 3-2. WASTEWATER FLOWS TO IAF DURING TEST PERIOD

Date	Time	Gallons per minute (gpm)
8-16-83	0830	495
	1040	510
	1400	250
8-17-83	0200	525
	0400	600
	0715	525
	0930	675
	1200	375
	1400	375
8-18-83	0300	488
	0530	675
	0600	450
	0730	525
	0900	450
	1000	375
	1100	413
	1130	420
	1200	375
	0300	413
8-19-83	0000	>700
	0100	525
	0200	563
	0300	600
	0400	525
	0500	450
	0600	450
	0700	488
	0800	525
	0900	563
	1000	600
	1100	>675
	1200	525

system to be drained regularly so that accumulated sludge can be removed. It was during this maintenance procedure that the upset occurred. The sour water treating system is shown in Figure 3-2.

As a result of the upset, abnormal operating procedures were also observed on Thursday, August 18. Plant operators determined a need to increase coagulant addition to the IAF. In order to accomplish this, one access door on the IAF was opened and coagulant fed manually to the unit. During this time, the plant air used to purge vapors from the IAF was turned off. This procedure was followed from approximately 07:30 to 10:10 on Thursday.

The amount of floating oil in the open basins gradually subsided as a result of remedial measures taken by the plant. Along with an increase of coagulant to the IAF, coagulant had also been directly added to the open bays on Wednesday in an effort to remove the floating oil.

4. LOCATION OF SAMPLE POINTS

The gaseous and water sampling locations used for the testing of the effluent treatment system serving the process waste waters are shown in Figures 4-1 through 4-3. The treatment system consisted of dual API separators feeding to an induced air flotation (IAF) tank. The inlet and outlet process water samples were taken from both the API and IAF systems. The gaseous sampling was performed on the ventilation air of the IAF tank.

The sample location for the ventilation air from the IAF unit was located in an adaptation configured in the ventilation air line. The pipe was disconnected prior to the off-gas blower feeding the IAF ventilation air to the process boiler. The locations of the gaseous sampling point and velocity measurement system are provided in Figure 4-3.

The IAF ventilation line adapted with the measurement system was a three-inch steel pipe. The steel pipe was fitted with a four-foot section of four-inch aluminum pipe, a volumetric totalizing flow meter, and four-inch plastic flexible ducting. The total flow in this line was routed through the flow volume measurement meter. Samples for determination of gaseous components were extracted through a ¼-inch hole in the aluminum piping prior to the meter.

The water sampling sites during these tests were the influent and effluent of the IAF system and the influent to the API separator system. The IAF influent was obtained from a sampling tap installed in the feed line from the API separator to the IAF tank. The API influent sample was obtained from a sampling tap in the oil water influent piping. The IAF outlet sample was obtained from a sampling tap after the IAF discharge pump.

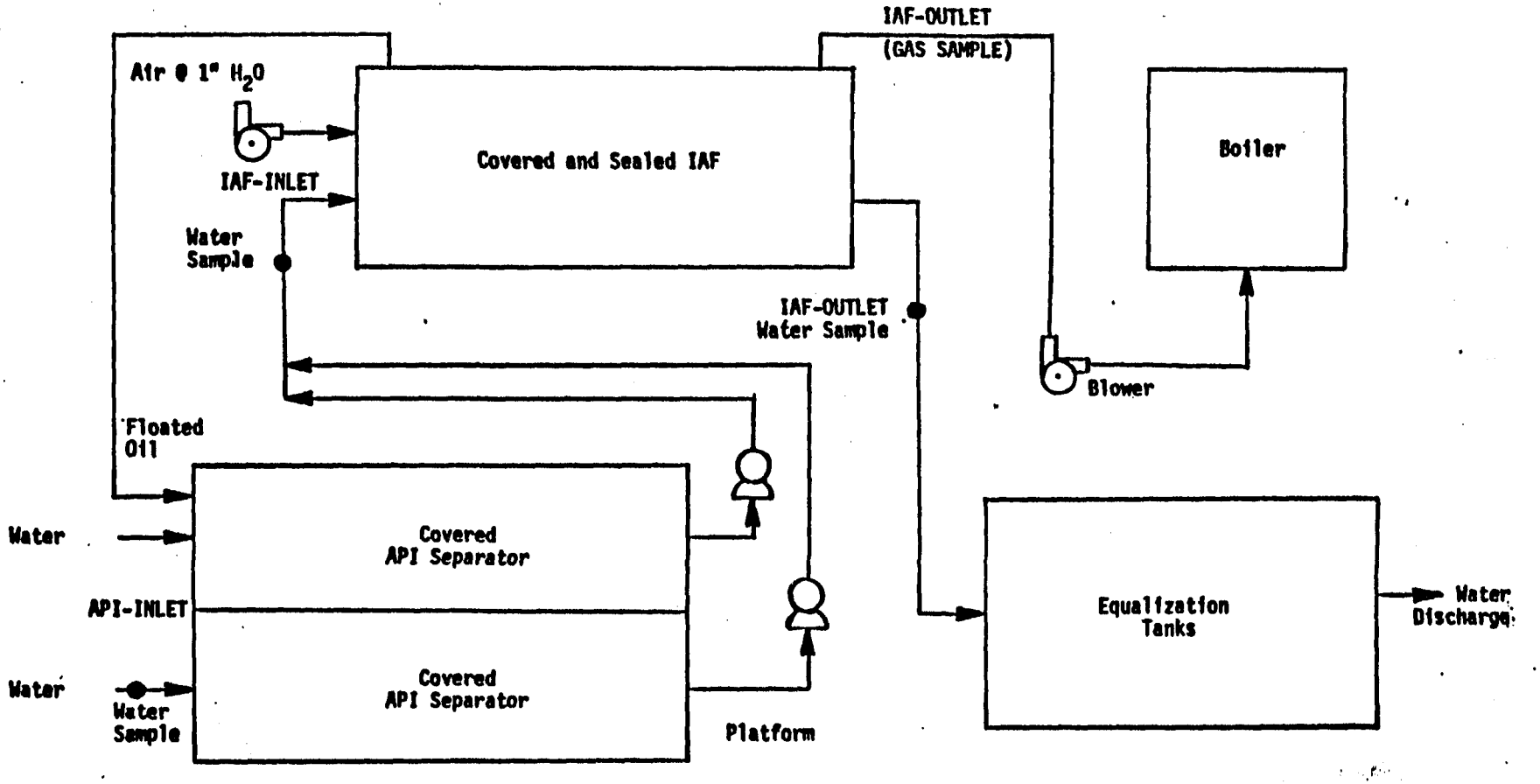


Figure 4-1. Sample locations at the wastewater treatment facility: Golden West - Santa Fe Springs, California.

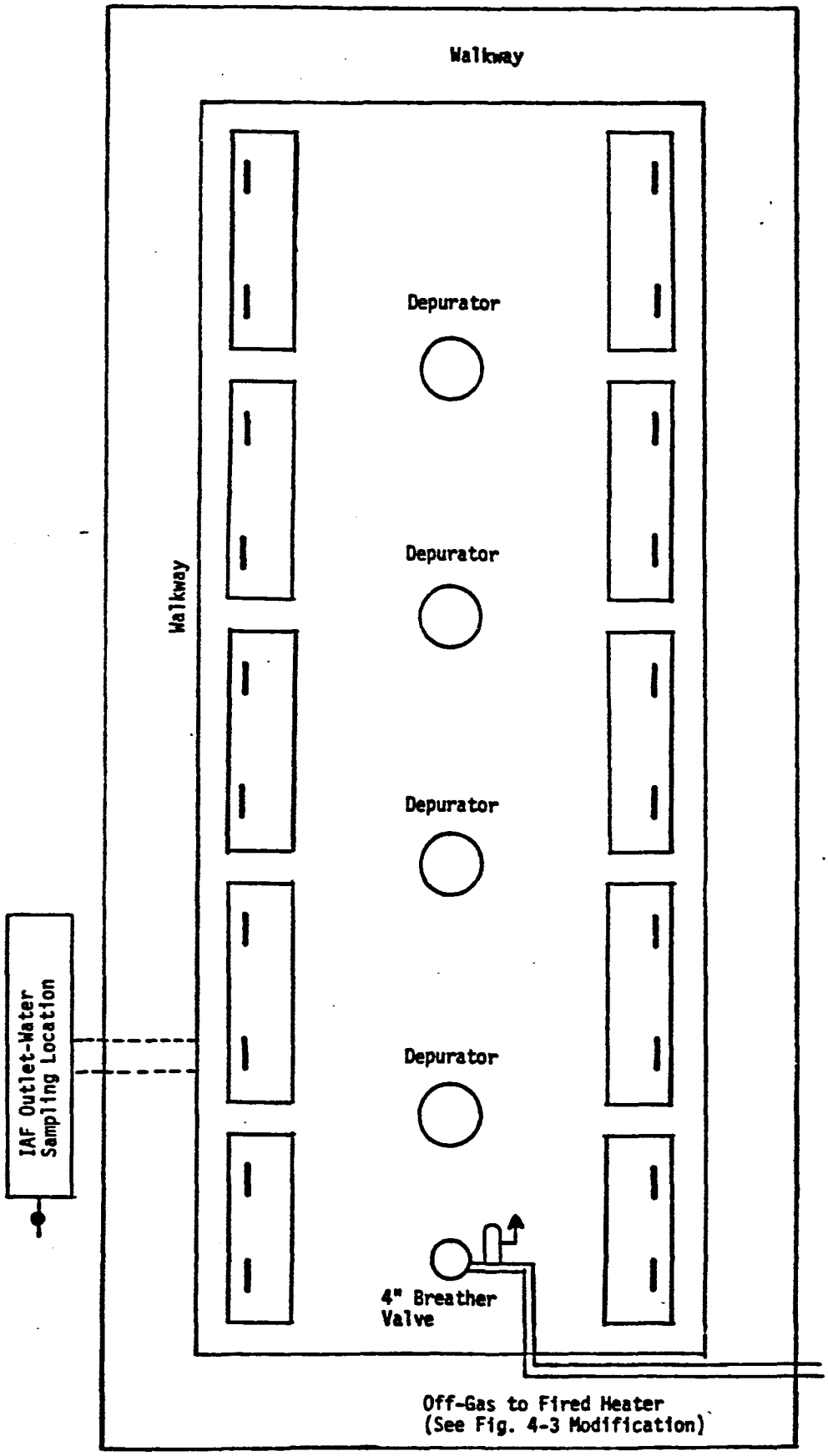


Figure 4-2. IAF tank system before off-gas line modification: Golden West, Santa Fe Springs, California

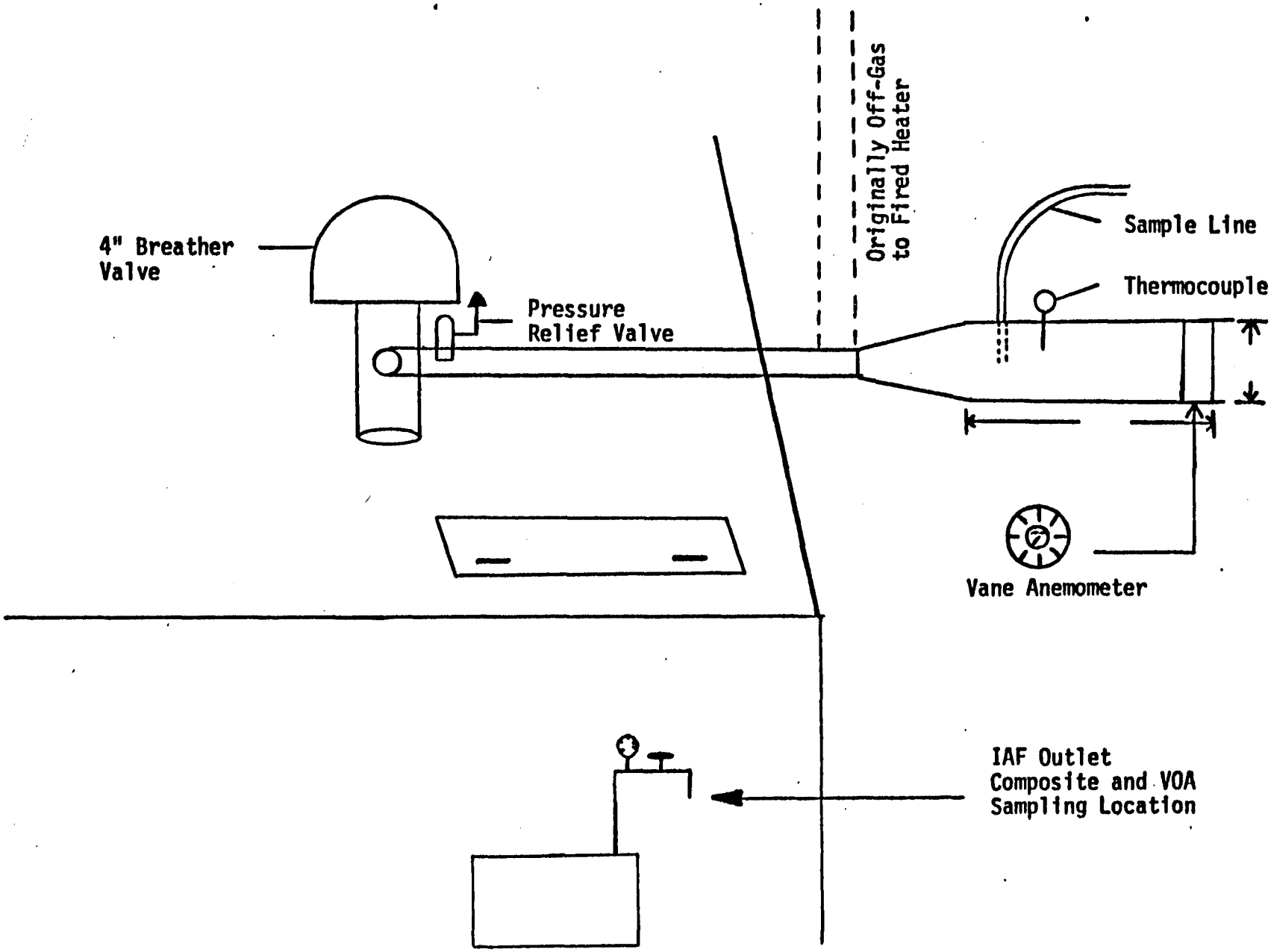


Figure 4-3. IAF measurement system modifications: Golden West, Santa Fe Springs, California.

5. SAMPLING AND ANALYTICAL PROCEDURES

The sampling and analytical procedures used to evaluate the gaseous and liquid streams are presented in this section. The methodologies are discussed separately as gaseous VOC methods (5.1), fixed gas analysis (5.2), gas flow measurement (5.3), liquid sampling (5.4), and liquid sample analysis (5.5).

5.1 GASEOUS VOC METHODS

Two procedures were used to measure the VOC content of the gaseous streams. EPA Method 18 was used to determine the general VOC species in the samples, and a procedure similiar to EPA Method 25A was used to measure the equivalent total hydrocarbon content of the streams.

5.1.1 EPA Method 18

"EPA Method 18. Measurement of Gaseous Organic Compound Emissions by Gas Chromotography", (promulgated October 18, 1983, Federal Register 48 FR 48328) was used to characterize the organic components in the streams tested. Samples were collected using the integrated bag technique of Method 18. Figure 5-1 illustrates the apparatus.

A clean 2.5 cubic foot TEDLAR® flexible bag was used for each run. The bags were cleaned by filling with dry nitrogen and venting the bag contents to the atmosphere until no background organics were detected by the analysis system. Prior to sampling the sampling apparatus and flexible bag were leak checked by evacuating each to 29" Hg vacuum and monitoring the pressure for 10 minutes. If a change of less than 1" Hg is observed, the components are judged leak-free. The sample probe, sample connecting tubing, and the sample bag were operated at ambient temperature. To prepare for sampling the vacuum source can was evacuated to -29" Hg. The system was then assembled and the sample probe was placed near the centroid of the duct to be sampled. Sampling was

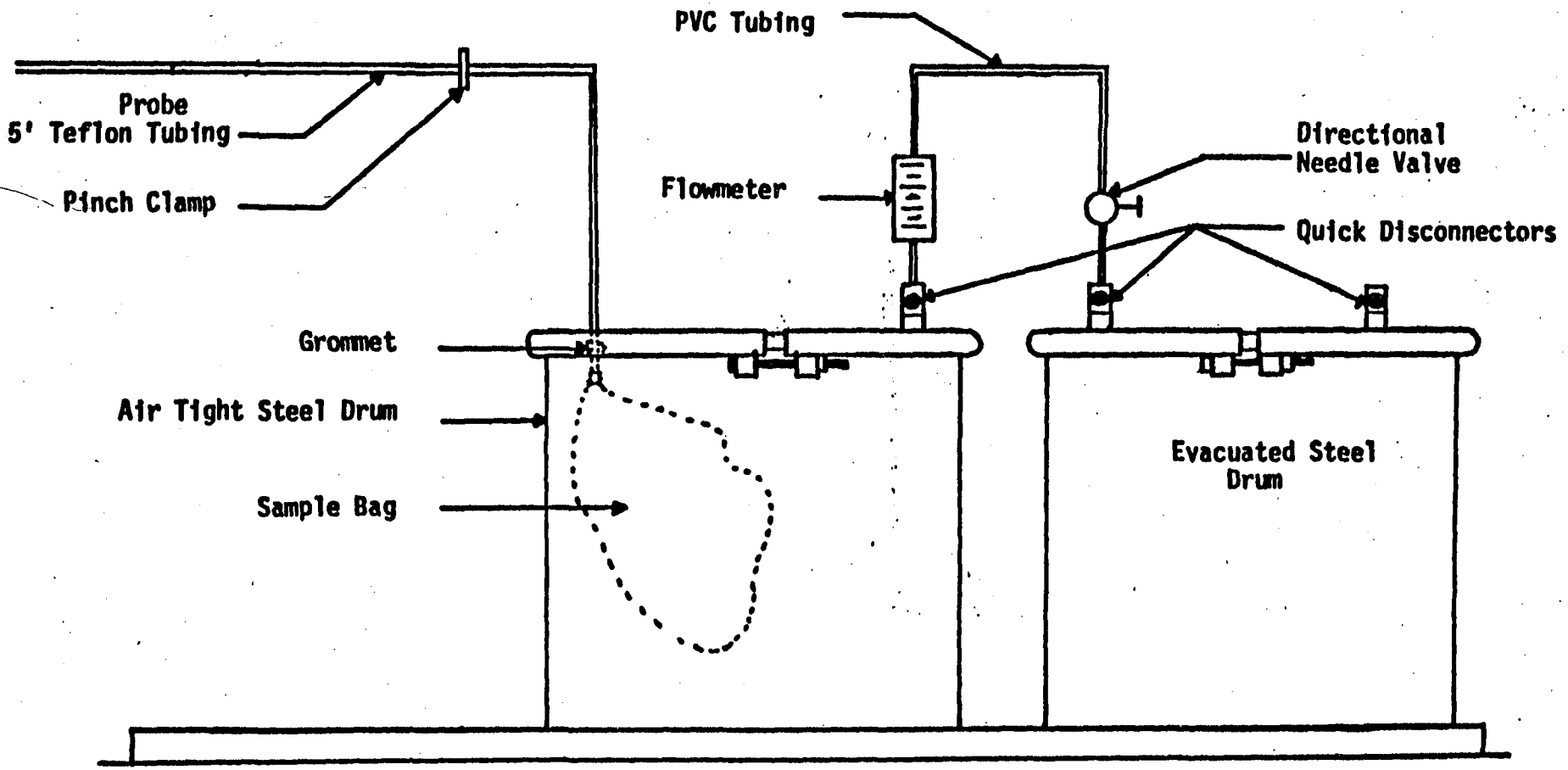


Figure 5-1. Gas Bag Sampling System.

started by opening the flow control valve and was maintained at a constant rate using the rotameter for about one hour. At the end of the sampling period, the flow valve was closed, the probe was disconnected, and the bag inlet was sealed. The sample bag was transported to the on-site mobile laboratory for analysis.

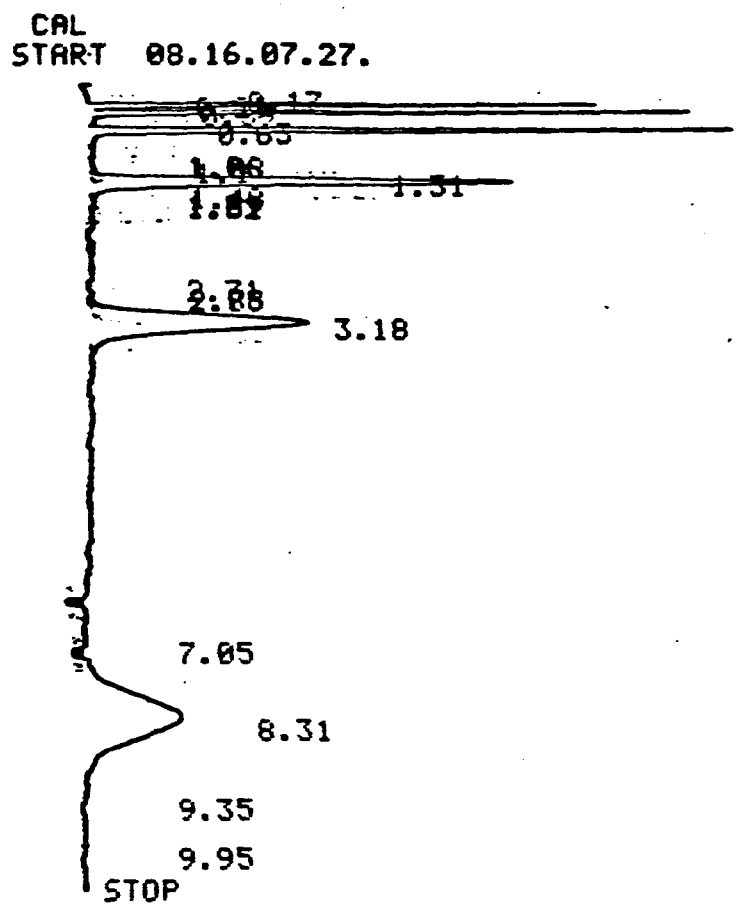
Two gas chromatograph systems with flame ionization detectors were used to analyze each sample. One system was used to separate and quantify low molecular weight parafins and olefins while the other system was used to measure aromatics and higher molecular weight components.

The system used to measure low molecular weight compounds (termed C_1-C_5 components) was a Shimadzu GC Mini 1 with a Shimadzu Chromatopac CRI-A digital integrator/recorder. The operating parameters were:

- Column: 6 ft x 1/8 in. I.D. stainless steel.
- Column support: Poracil C.
- Column temperature/program: 35°C/constant.
- Sample loop size/temperature: 1 ml/ambient.
- Carrier gas/flow: He/50 ml/min.

A calibration gas mixture containing known concentrations of methane, (15.1 ppm) ethane (14.6 ppm), propane (15.6 ppm), butane (15.2 ppm), and pentane (15.6 ppm) in nitrogen was used to obtain a area factor and retention time for each of these compounds. Figure 5-2 presents an example of a GC/FID calibration run for C_1-C_5 speciation. During sample analysis, the peaks near these retention times were grouped as the nearest carbon number, and the concentration was calculated using the corresponding calibration factor for that carbon number. Figure 5-3 presents an example of GC/FID analysis runs for C_1-C_5 speciation at the IAF sample location.

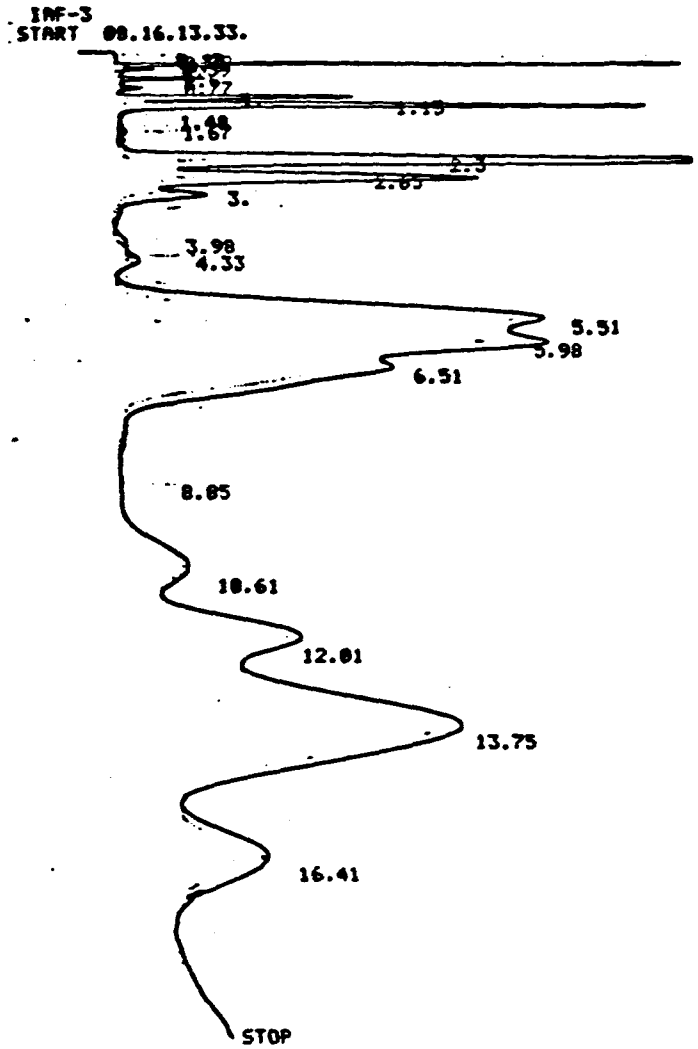
The GC/FID analysis run for C_1-C_5 speciation (Figure 5-3) provides an illustrative example of problems in the analytical procedures. Standards were not available to provide elution time reference for all peaks identified by the GC/FID with areas. Therefore, the total organic concentration from the analysis was a sum of all the peaks and not just the identified peaks. Another problem was the slight variance of the elution time during the test day. The GC/FID operator noted an increase



C-R1A
SMPL # 00
FILE # 7
REPT # 118
METHOD 44

#	NAME	TIME	CONC	MK	AREA
		0.17			43
		0.29		V	1167
1	METH	0.39	0	V	1362
2	ETHA	0.63	0	V	1955
		1.31		V	2652
		2.78			27
		3.18		V	3443
		7.05			232
		8.31		V	4556
		9.35		T	25
		9.95		T	94
	TOTAL		0		15560

Figure 5-2. Example of GC/FID calibration for C_1-C_5 speciation.



C-R1A
SMPL # 08
FILE # 7
REPT # 127
METHOD 44

#	NAME	TIME	CONC	MK	AREA
		0.13			107
		0.19		V	16
		0.2		V	16
		0.22		V	17
		0.3		V	6734 C1
1	METH	0.39	0	V	931 C2
2	ETHA	0.6	0	V	1887 C3
		0.77		V	913
3	PROP	1.	0	V	7051
		1.15		V	19022 C4
		1.67		T	366
4	BUTA	2.3	0	V	52614
		2.65		V	29131 C5
		3.		V	8099
		3.98			1117
		4.33		V	2987
5	PENT	5.51	0	V	95587
5	PENT	5.98	0	V	91529
		6.51		V	68406 C6
		8.85		T	103
		10.61		V	26906
		12.01		V	68110
		13.75		V	227161
6	HEXA	16.41	0	V	88746
	TOTAL		0		797575

Figure 5-3. Example of GC/FID analysis on IAF ventilation air - gas bag sample for C_1-C_5 speciation.

in elution time with the increase in field lab temperature. The operator noted the temperature differential in the field laboratory across a test day on the Chromatopac. The analysis peak times were adjusted accordingly.

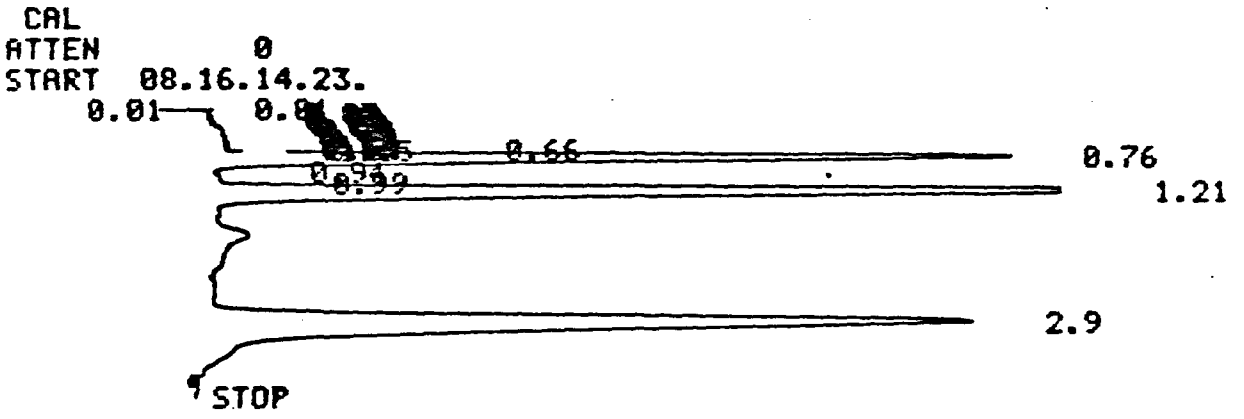
The system used to measure aromatic and higher molecular weight compounds (termed semi-volatile) was a Shimadzu GC Mini II equipped with a Shimadzu Chromatopac integrator. The operating parameters were:

- Column: 6 ft x 1/8 in. I.D. stainless steel.
- Column support: OV-1 on 80/100 Supelco.
- Column temperature/program: 25°C/constant.
- Sample loop size/temperature: 1 ml/225°C.
- Carrier gas/flow: He/20 ml/min.

A calibration mixture of 49.8 ppm benzene and 49.9 ppm m-xylene in nitrogen was used to determine calibration factors and retention times for these two compounds. Qualitative gaseous standards were prepared from liquid hexane, heptane and toluene were used to determine the retention time for these compounds. Figure 5-4 presents an example of a GC/FID calibration run for C_6-C_9 speciation. During sample analysis, hexane, heptane, benzene and toluene were expressed as equivalent benzene concentrations and C_8 and higher components were expressed as m-xylene equivalent concentrations. Figure 5-5 presents an example of GC/FID analysis runs for C_6-C_9 speciation at the IAF sample location.

5.1.2 EPA Method 25A

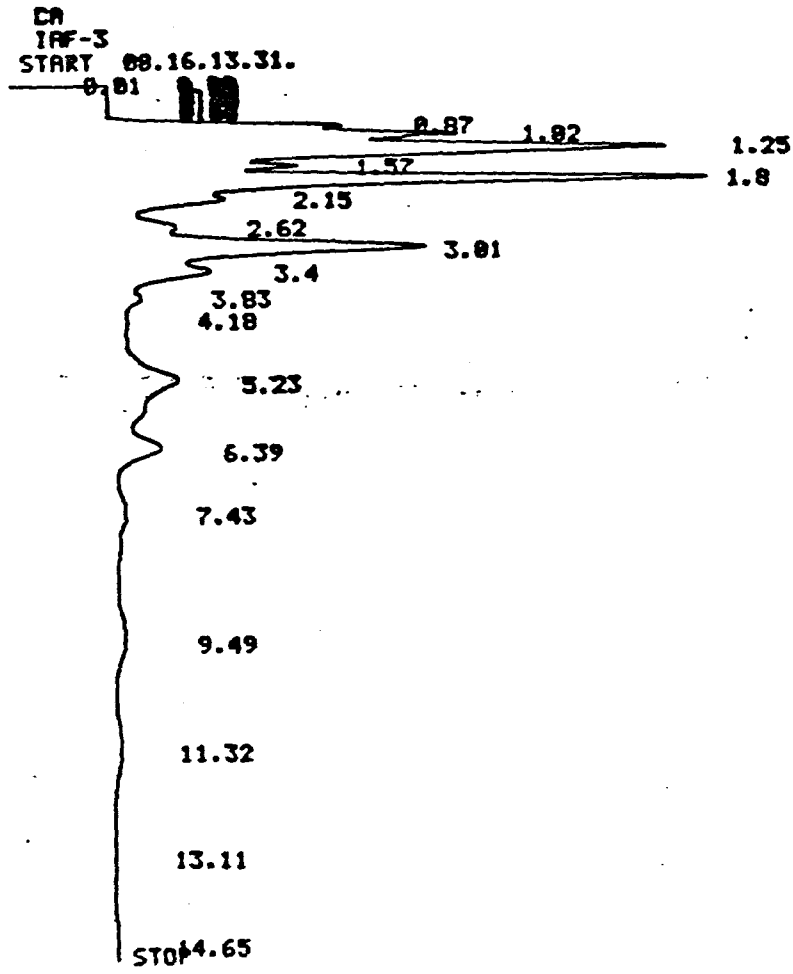
Procedures similiar to those described in EPA Method 25A (Federal Register 48 FR 37595) were used to continuously measure the total hydrocarbon concentration in the gaseous streams tested. A Beckman Model 402 flame ionization analyzer was used at the IAF sample locations. The sample probes were placed near the centroid of the duct to be sampled. A continuous sample flow was maintained through heated Teflon® sampling lines. The instrument operating parameters were:



C-R1A
SMPL # 00
FILE # 2
REPT # 246
METHOD 44

#	NAME	TIME	CONC	MK	AREA
1	C1-C5	0.76	0	V	5792
		1.21		V	8190
		2.9			8280
	TOTAL		0		22263

Figure 5-4. Example of GC/FID calibration for C_6-C_9 speciation.



C-R1A
SMPL # 08
FILE # 2
REPT # 243
METHOD 44

#	NAME	TIME	CONC	MK	AREA	
		0.87		V	80538	
		1.02		V	156277	hex
3	BENZ	1.25	0	V	356181	benz
4	HEPT	1.57	0	V	73398	
		1.8		V	341358	tol
5	TOLU	2.15	0	V	66129	
		2.62		V	47881	
		3.01		V	246554	m-xyl
		3.4		V	79865	o-xyl
6	-M XYL	3.83	0	V	26431	
7	OOXYL	4.18	0	V	13649	
		5.23		V	156822	
		6.39		V	63788	
		7.43		V	48513	
		9.49		V	42567	
		11.32		V	23872	
		13.11		V	2488	
		14.65			1879	
	TOTAL		0		1819139	

Figure 5-5. Example of GC/FID analysis on IAF ventilation air - gas bag sample for C_6-C_9 speciation.

Site: IAF ventilation air and carbon drum

- Analyzer: Beckman Model 402.
- Serial #: 1001303.
- Fuel Pressure: 25 PSI.
- Sample Pressure: 3.0 PSI.
- Air Pressure: 16 PSI.
- Sample line length/approximate temperature: 25 feet/ambient.

The analyzers were operated continuously 24-hours per day. The analyzers were equipped with strip chart recorders for data reduction. The instruments were calibrated with compressed gas standards of propane in a balance of air.

The calibration gas standards were supplied by Scott Specialty Gases and certified to within ±2 percent of the labeled calibration gas values. The calibration gas standards used at Golden West are, listed in ppm as propane: 49.9, 100.1, 500.5, 1002.5, and 4010.1.

The initial calibration prior to commencing a test series at a test location, or follow-up calibration prior to re-commencing a test series after an instrument shut-down, included the following calibration sequence. First a trial sampling of the source stream would indicate the appropriate concentration range for which the instrument would be operated. Second, the initial calibration of the instrument on this pre-determined scale included introducing zero gas and the high-level calibration gas separately to the sample manifold. The output was then adjusted to the appropriate levels. No instrument adjustments were made after this time. Third, a periodic response check was performed by introducing the zero and high level calibration gas with no adjustments. A response within ±1 percent of span value was required or recalibration would have been performed. Fourth, a linearity check was performed on the instrument span range by introducing mid-level and low-level calibration gases. The difference between the measurement system responses and the predicted response were recorded. The differences were assured to be less than five percent of the respective calibration gas values before the measurement system was placed on-line for monitoring.

The operational parameters and calibration gas standards used at the IAF sample location were as follows:

- Instrument: Beckman 402.
- Scale: 0-10,000 ppm.
- Low-level calibration gas standard: 500.5 ppmv as C_3H_8.
- Mid-level calibration gas standard: 1002.1 ppmv as C_3H_8.
- High-level calibration gas standard: 4010.0 ppmv as C_3H_8.

A monitor system response time check was performed at the IAF sample location. The check was performed by introducing the high-level calibration gas at the inlet to the sample line feeding the measurement system. The time interval was measured for the analyzer to respond by 95 percent of the calibration gas value. The short sample lines gave response time of 17 seconds and is within the allowed limit of 30 seconds.

Zero and span drift determinations were made during and after each test period. The frequency of drift checks were determined by the operational status of the analyzer and total length of the test. During the initial operation of the analyzer, after the measurement system had been powered down, the FIA required frequent drift checks (one to two hours) for maintaining the drift values below the specified three percent limit. A complete calibration sequence was completed if a drift check demonstrated the necessity. After the frequent drift checks verified the calibration stability of the measurement system, the drift checks were performed three times during the 12-hour test day period (0600, 1200, 1700). Test periods of before a 12-hour instrument operation period required the two-hour drift check frequency.

Figure 5-6 presents an example of a calibration check at the IAF sampling location with a recalibration required. The sequence was initiated by introducing zero, high, and mid-calibration gases separately. The upward drift at the three levels was approximately one percent. Therefore, the zero and high standards were re-introduced and analyzer adjustments made. Next, a linearity check is performed with the mid standard (no adjustment) and the sample reconnected for monitoring.

The calibration gas level reintroduced for the drift check was determined by the sample measurement levels. The IAF sample locations

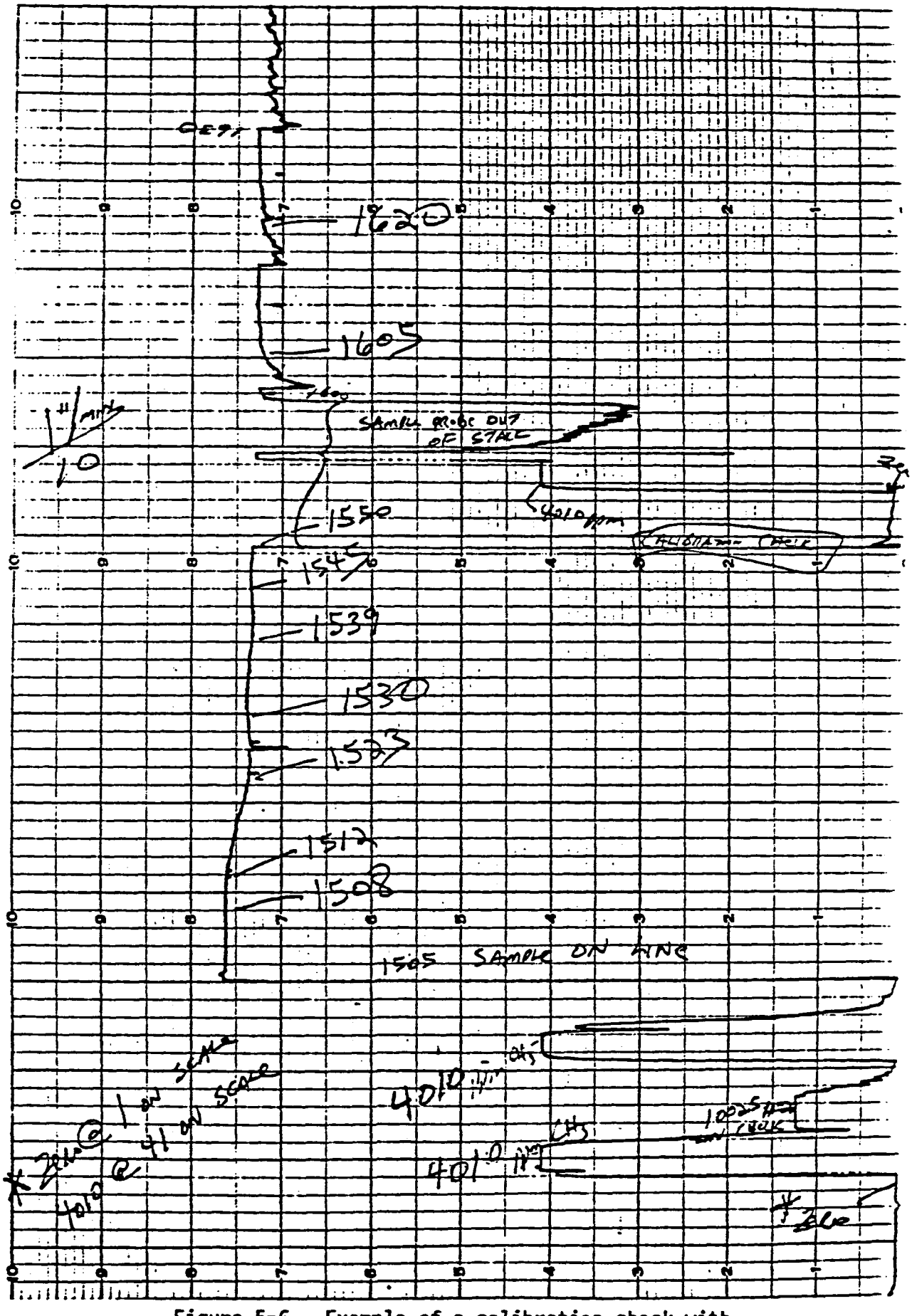


Figure 5-6. Example of a calibration check with a recalibration required.

ranged from 6000-7500 ppm and the 4010.0 ppmv as C_3H_8 was used for the span drift check.

The continuous monitor data were reduced by determining the average organic concentration measured into ppmv as propane. Direct computation of the recorded stripchart outputs was applicable because the high-level standard was calibrated on the same span value as the recorder. The IAF measurement system was calibrated on the 0-10,000 ppm scale. Therefore, the IAF calibration was performed with 40 percent of scale being equivalent to the 4010 ppmv as propane.

The measured concentrations are presented on a ppmv as propane equivalent. The one-hour concentration averages were calculated from direct output readings at five-minute intervals. The average results were calculated on the hour. Hour periods with drift checks and calibration were normalized to the hour from the partial segments of the hour.

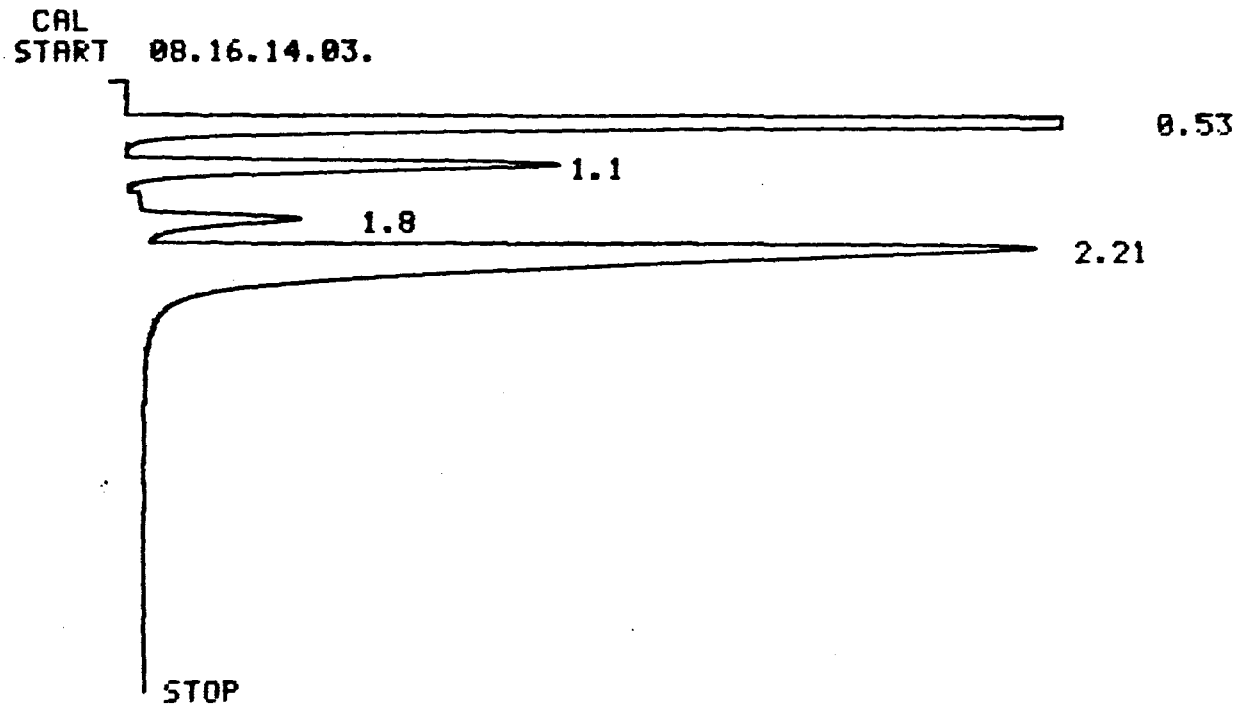
5.2 PERMANENT GAS ANALYSIS

A gas chromatograph equipped with a thermal conductivity detector was used to analyze each bag sample collected for VOC analysis to determine the nitrogen, oxygen, and carbon dioxide content. This procedure was intended to characterize the balance gas constituents and is allowed in Section 1.2 of EPA Method 3 (42 FR 41768) for this purpose.

The chromatograph used was a Shimadzu GC-3BT with a Shimadzu Chromotopac integrator. The operating parameters were:

- Column: 6 ft x 1/8 in. I.D. stainless steel.
- Column support: 60/80 molecular sieve.
- Column temperature/program: 105°C/constant.
- Sample loop size/temperature: 2 ml/35°C.
- Carrier gas/pressure: He/3 PSI.

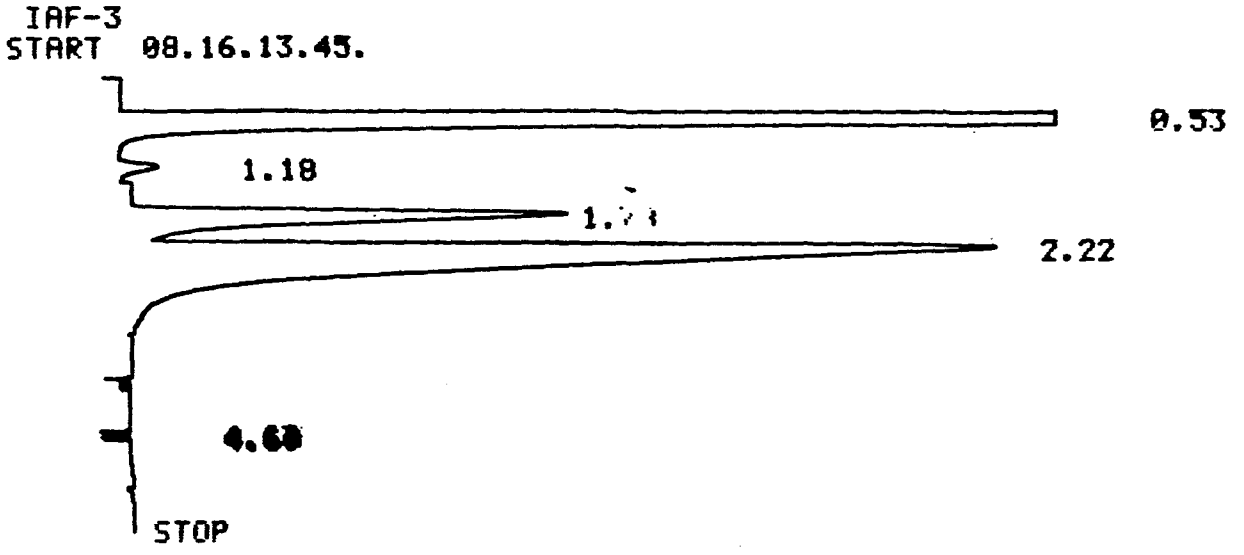
The chromatograph was calibrated with a cylinder standard of Scotty II-Mix containing (by volume) 14.8 percent CO_2, 7.07 percent O_2, 78.13 percent N_2 with a ±2 percent certification. Figure 5-7 provides an example of GC/TCD calibration and Figure 5-8 provides an example of a GC/TCD analysis run for the balance gas constituents in the IAF unit #1 sample.



C-R1A
SMPL # 00
FILE # 4
REPT # 1597
METHOD 44

#	NAME	TIME	CONC	MK	AREA
1	AIR	0.53	98.07		47427
2	CP-2	1.1	14.726		8973
3	O-2	1.8	6.0846		3330
4	N-2	2.21	75.4922	V	37134
	TOTAL		194.373		96865

Figure 5-7. Example of GC/TCD calibration for stationary gas analysis.



C-R1A
SMPL # 00
FILE # 4
REPT # 1595
METHOD 44

#	NAME	TIME	CONC	MK	AREA
1	AIR	0.53	110.0757		53233
2	CP-2	1.18	0.9662		588
3	O-2	1.78	18.3754		10058
4	N-2	2.22	72.5525	V	35688
	TOTAL		201.9699		99569

Figure 5-8. Example of GC/TCD analysis on equalization tank #2 gas bag sample for stationary gases.

5.3 GASEOUS VOLUMETRIC FLOW MEASUREMENT

At the induced air flotation unit vent a modification of EPA Method 2A (Federal Register Vol. 48, No. 247, December 22, 1983) was used. The flow rate at this site was small and the pressure insufficient for driving a meter recommended by Method 2A. Unsuccessful attempts were made using a turbine-type meter with a 9000 cubic foot per hour rating. The system that proved useable was a fabricated meter based on a four-inch diameter anemometer housed in a section of duct with the same nominal diameter as the aneometer:

A jewelled anemometer was used at the IAF sample location for measuring the velocity through a four-inch adaption between the IAF ventilation air and a pseudo-exhaust (not an ambient exhaust in original configuration). Figure 4-3 provides a schematic representation of the velocity measurement system.

The anemometer was calibrated by the manufacturer (Davis Instrument Mfg.) and the calibration/correction data is provided in Appendix A. No in-house calibration was performed since this was the first use of the anemometer.

The operation of the IAF unit was constant in flow rate and direction. Initial measurements at short time intervals during the test period exemplified the constant flowrate. Therefore, four to six-hour time intervals were sufficient in cumulating a total volumetric measurement over a known time period.

5.4 LIQUID SAMPLE METHODS

Liquid process samples were collected from sample taps used by the refinery for process quality control. Two types of samples were collected and were termed "void of air" (VOA) and "composite".

The void of air samples were collected by completely filling a 40 mL bottle with a grab sample. These bottles are fitted with a special cap to eliminate air bubbles from the sample. The composite samples were collected by combining three to four equal volume grab samples into a one gallon amber bottle.

Both sample types were taken from a process stream flowing in a pipe, through a sample line which was purged prior to sample collection. The samples were stored on ice in insulated containers after collecting,

and during shipment to the TRW facility at the Research Triangle Park, North Carolina for analysis. The bottles were prepared by the following cleaning procedures:

a) strong soap solution;

b) liberal tap water rinse;

c) nitric acid, 15 percent v/v;

d) distilled water rinse;

e) methanol rinse;

f) methylene chloride rinse; and

g) drying a clean, hot air stream or placing in an oven at 40°C (140°F).

After the containers have been cleaned, dried and capped, they are stored in boxes to prevent spurious contamination.

5.5 LIQUID SAMPLE ANALYSIS METHODS

5.5.1 Total Organic Carbon (TOC)

This method is applicable to the measurement of organic carbon in drinking and surface waters as well as domestic and industrial wastes.

5.5.1.1 Summary of Method. Organic carbon in a sample is converted to carbon dioxide (CO_2) by photochemical oxidation. The CO_2 is measured to determine the total organic carbon. The sample is initially purged by vacuum to remove inorganic carbon. Sample inorganic carbon is eliminated or must be compensated for because it is usually a large part of the total carbon. The instrument is calibrated versus a standard solution of potassium hydrogen phthalate (KHP).

5.5.1.2 Interferences/Quality Control. Removal of carbonate and bicarbonate by acidification and purging with nitrogen or other inert gas can result in the loss of volatile organic substances. Volatiles also can be lost if the samples are allowed to heat up.

Repeatability of replicate injections can be effected by non-homogeneity of samples. This can occur if large carbon containing particulate matter is not representatively collected in the sample injection syringe. It is also necessary to collect and maintain the samples in bottles with no head space so as to minimize the volatilization

of organic components. This phenomenon is apparent after the TOC analysis of theoretically identical samples in which one was collected in a VOA bottle (no head space) and another collected in a larger sample bottle only half to three-quarters full. Repeatability and representativeness can be improved by homogenizing (by mixing) the samples prior to analysis.

The precision measurement based upon repeated injection of three randomly selected samples appears to be a function of the concentration when measured on the basis of the standard deviation. The standard deviations in one case for two of the samples cannot be considered to be equal (at the 95% level of significance), but there appears to be no difference between the standard deviations when comparing one of the first two with the third. When compared on the basis of the relative standard deviation, RSD, (or % RSD), the precision for all three measurements appears to be the same.

The accuracy of the technique is best represented here by injecting a known volume of the calibration standard and comparing the results to the theoretical value. In this case, the highest standard used to calibrate was a 100 mg/L (100 ppm) solution of KHP.

The accuracy measurement is based on an in-house standard and indicates about a 9% positive bias (or accuracy), based on the mean of the measurements. However, a statistical hypothesis test that the bias is zero would be accepted at the 95% level of confidence. Stated differently there is a 95% probability of being correct if the data does not show a significant difference from zero.

All samples were diluted as necessary to fall within the limits of the calibration.

5.5.2 Chemical Oxygen Demand (COD)

This test method is considered an independent measurement of the organic matter in a sample. The Chemical Oxygen Demand (COD) method determines the quantity of oxygen required to oxidize the organic matter in a waste sample, under specific conditions of oxidizing agent, temperature, and time.

5.5.2.1 Summary of Method. Organic and oxidizable inorganic substances in the sample are oxidized by potassium dichromate in 50% sulfuric acid solution at reflux temperature. Silver sulfate is used as a catalyst and mercuric sulfate is added to remove chloride interference.

The excess dichromate is titrated with standard ferrous ammonium sulfate, using orthophenanthroline ferrous complex as an indicator.

5.5.2.2 Interferences/Quality Control. Volatile straight-chain aliphatic compounds are not oxidized to any appreciable extent. This limitation occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight-chain aliphatic compounds are oxidized more effectively when silver sulfate is added as a catalyst. However, silver sulfate reacts with the halides to produce precipitates that are only oxidized partially. This can be partially overcome by adding mercuric sulfate to complex the halides prior to the reflux step.

The replicated chemical oxygen demand readings are given in Table 5-1. Seven samples were replicated and the means, standard deviations, and coefficients of variation for the COD readings are given in Columns 4, 6, and 8, respectively in the table. Assuming that the coefficient of variation of the chemical oxygen demand (COD) readings should remain constant, the pooled estimate of the coefficient of variation is 0.0435, or 4.4% and is a good measure of the precision.

5.5.3 Oil and Grease

This method includes the measurement of fluorocarbon-113 extractable matter from industrial and domestic wastes. It is applicable to the determination of relatively nonvolatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter.

5.5.3.1 Summary of Method. The sample is acidified to a low pH (<2) and serially extracted with fluorocarbon-113 in a separatory funnel. The solvent is evaporated from the extract and the residue weighed on an analytical balance.

5.5.3.2 Interferences/Quality Control. Fluorocarbon-113 has the ability to dissolve not only oil and grease but also other organic substances. No known solvent will selectively dissolve only oil and grease. Solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatization. Significant portions of petroleum distillates from gasoline through No. 2 fuel oil are lost in the process. In addition, heavier residuals of crude oils and heavy fuel oils contain a significant percentage of residue-type materials that are not soluble in fluorocarbon-113.

Table 5-1. REPLICATED COD AND OIL AND GREASE MEASUREMENTS

TRW Sample #	COD mg/L	O & G mg/L	Means COD mg/L	Means O & G mg/L	Standard Deviation COD mg/L	Standard Deviation O & G mg/L	Coefficient of Variation COD	Coefficient of Variation O & G
4957	2968	491	3238	513.0	381.8	31.1	0.1179	0.0606
	3508	535						
4958	4106	453	4119	444.7	102.6	7.23	0.0249	0.0163
	4024	440						
	4228	441						
4960	2155	382	2135	379.0	29.0	4.24	0.0136	0.0112
	2114	376						
4961	1748	133	1748	133.5	0.0	0.71	0.0000	0.0053
	1748	144						
4962	1545	125	1565	115.0	20.0	18.2	0.0218	0.1582
	1585	94						
	1565	126						
4971	1240	110	1271	109.5	43.1	0.71	0.0340	0.0065
	1301	109						
4973	1911	123	1891	121.5	29.0	2.12	0.0153	0.0175
	1870	120						

Replicated oil and grease (O & G) readings for seven samples are shown in Table 5-1. Sample means, standard deviations, and coefficients of variation are shown in Columns 5, 7, and 9, respectively. Pooling the coefficients of variation for oil and grease gives a precision of 7.9, or almost double the precision of the COD readings.

5.5.4 Total Chromatographable Organics (TCO)/Hydrocarbon Speciation

This method is applicable for the measurement of hydrocarbons in surfaces waters, domestic and industrial wastes.

5.5.4.1 Summary of Method. The analysis for TCO was performed by gas chromatography with flame ionization detection. Component speciation was done by separation with a fused silica capillary (0.25 mm), GC column (SPB-1 boiling point column). The reported values are in milligrams per liter of sample, and is a total integrated value representing hydrocarbons ranging between C_5 and C_{30}.

For additional breakdown, i.e., hydrocarbon speciation, the resulting chromotography was broken down into C (toluene) through C_{30} hydrocarbons. The values in milligrams per liter were calculated using average response factors of C_7-C_{11}, C_{11}-C_{16} and C_{17} to C_{25} hydrocarbons. Due to the reduced response on a FID of C_{17} to C_{25} hydrocarbons as compared to C_7-C_{11} high values of some C_{17}-C_{25} compounds were found.

Each sample was prepared by extracting a 500 ml aliquot with methylene chloride both at an acidic and basic pH, combining the methylene chloride extracts, and then reducing the solvent to a final volume of 25 ml. Each sample was spiked with an internal spike to check recovery.

5.5.4.2 Interferences/Quality Control. The sample is serially extracted with methylene chloride and concentrated to provide sufficient hydrocarbons for analysis. The concentration step results in the loss of short-chain hydrocarbons and simple aromatics (BP < 100°C) by volatilization. In addition, the extraction partition coefficient for certain compounds does vary. For a measure of extraction efficiency, each sample and control samples (distilled organic free water) were spiked with Napthalene-d_8 which resulted in recoveries between 75 and 85 percent.

5.5.5 Purge and Trap (Volatile Organic Analysis)

The volatile organics in water were qualitatively identified by utilizing EPA Method 624 with mass spectral identification. After

examination of several representative samples, each water sample was quantitated by purge and trap with GC/FID.

5.5.5.1 Summary of Method. The GC/MS analysis was performed on a Finnigan 4000 with an INCOS data system. A Tekmar purge and trap apparatus was used according to EPA Method 624. The GC column used was a 6 ft x 1/8 in stainless steel packed with 0.2% CW 1500 on 60/80 Carbopack C. Oven conditions were 15°C programmed to 190°C at 10°C/min and held for 25 minutes. A 5 ml aliquot of each sample was taken for analysis and spiked with 750 ng Bromofluorobenzene (BFB) for an internal standard. Comparison by identification by GC/MS was done by spectral library searches and comparison with known standards (Figures 5-9 and 5-10). Quantitative analysis was obtained by GC/FID (Figures 5-11 and 5-12) using the same identical chromatography conditions as employed with GC/MS qualitative analysis runs.

5.5.5.2 Interferences/Quality Control. Contamination can occur whenever high-level and low-level samples are analyzed sequentially. When utilizing GC/FID detection only, co-eluting peaks can give a positive bias to values obtained for the components of interest.

Data quality techniques utilized for these analyses included the following:

1. A complete page of the system following a high level VOA sample.
2. Bromoflurobenzene (BFB) was used as an internal standard in all samples and control standards. In addition, benzene and toluene were quantitatively based on their respective response values to BFB.
3. When evidence of co-eluting was detected, values were not reported for selected compounds.

Table 5-2 gives concentration and quality parameters for an in-house standard and replicated results for TRW Sample No. 4973. BFB is bromofluorobenzene and was spiked into the in-house standard sample and the three replicated samples at the same concentration. The accuracy is estimated as the percent bias the mean of the three BFB readings is from the in-house standard, and is calculated to be about 52 percent. Precision is estimated as the pooled coefficient of variation for all the compounds (including BFB) and is calculated to be 9.6 percent. The sample here was not filtered before the replicated samples were drawn.

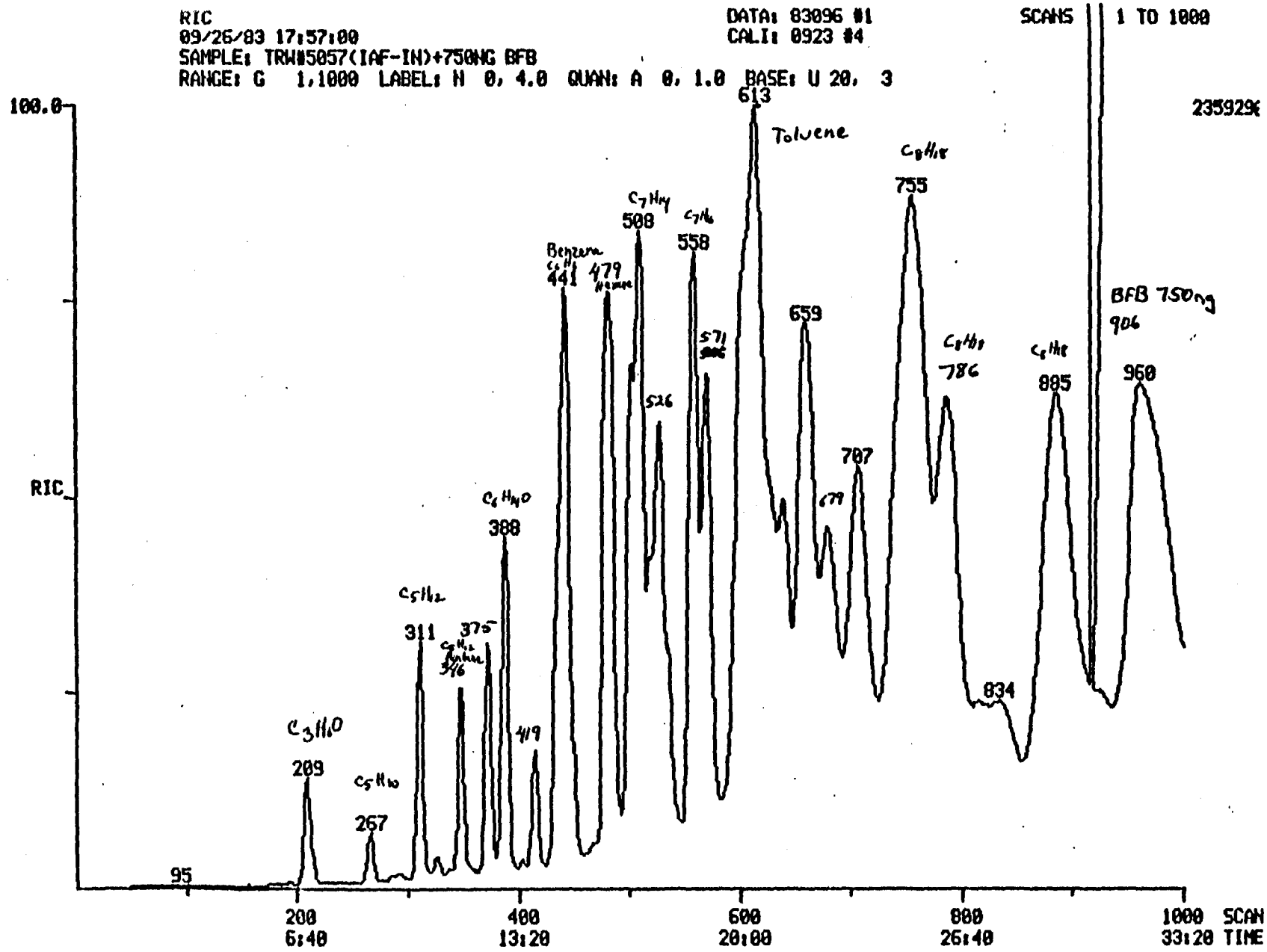


Figure 5-9. Mass spectrometer qualitative analysis by purge and trap, sample no. IAF-INLET-VOA-0740.

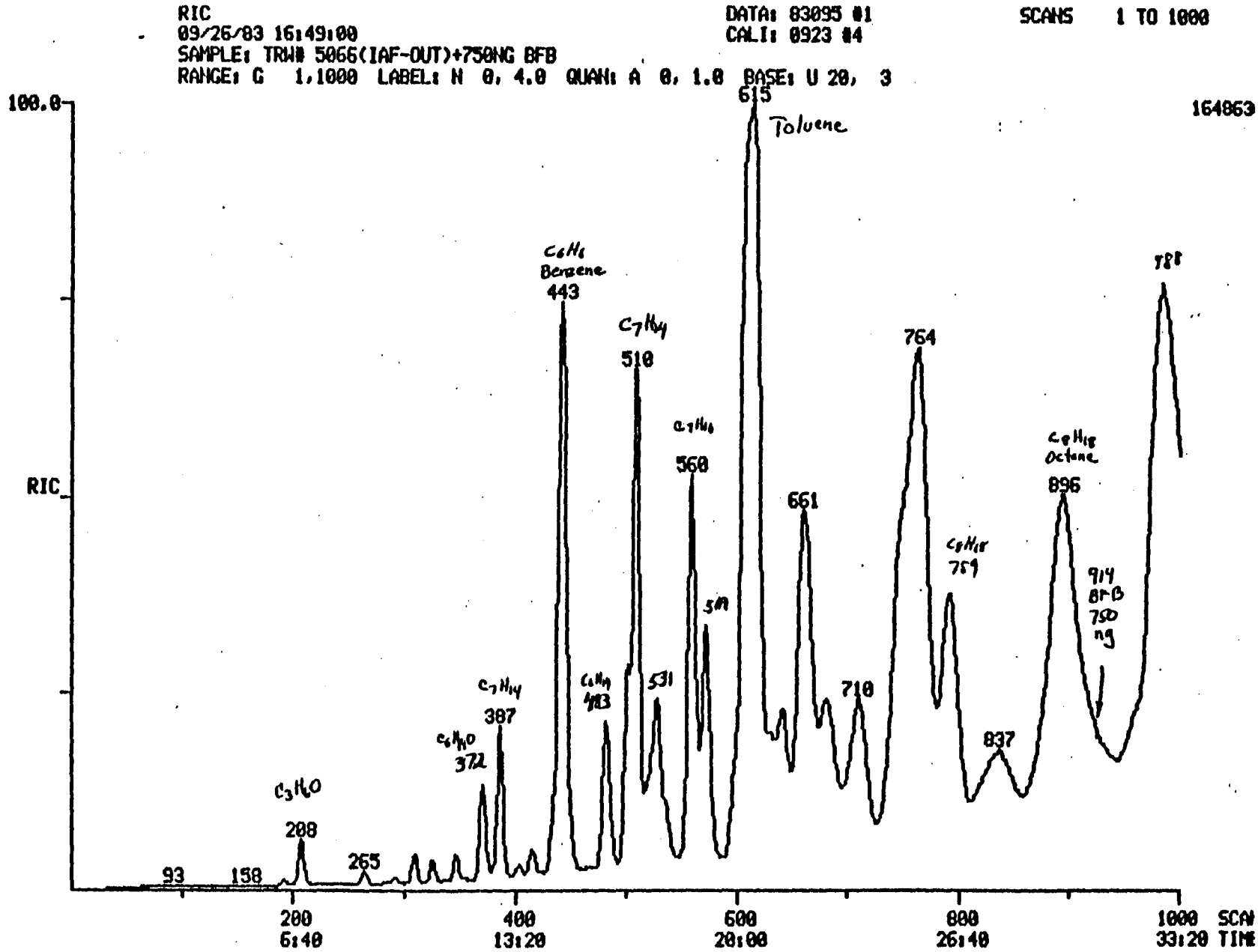


Figure 5-10. Mass spectrometer qualitative analysis by purge and trap, sample no. IAF-OUT-VOA-0740.

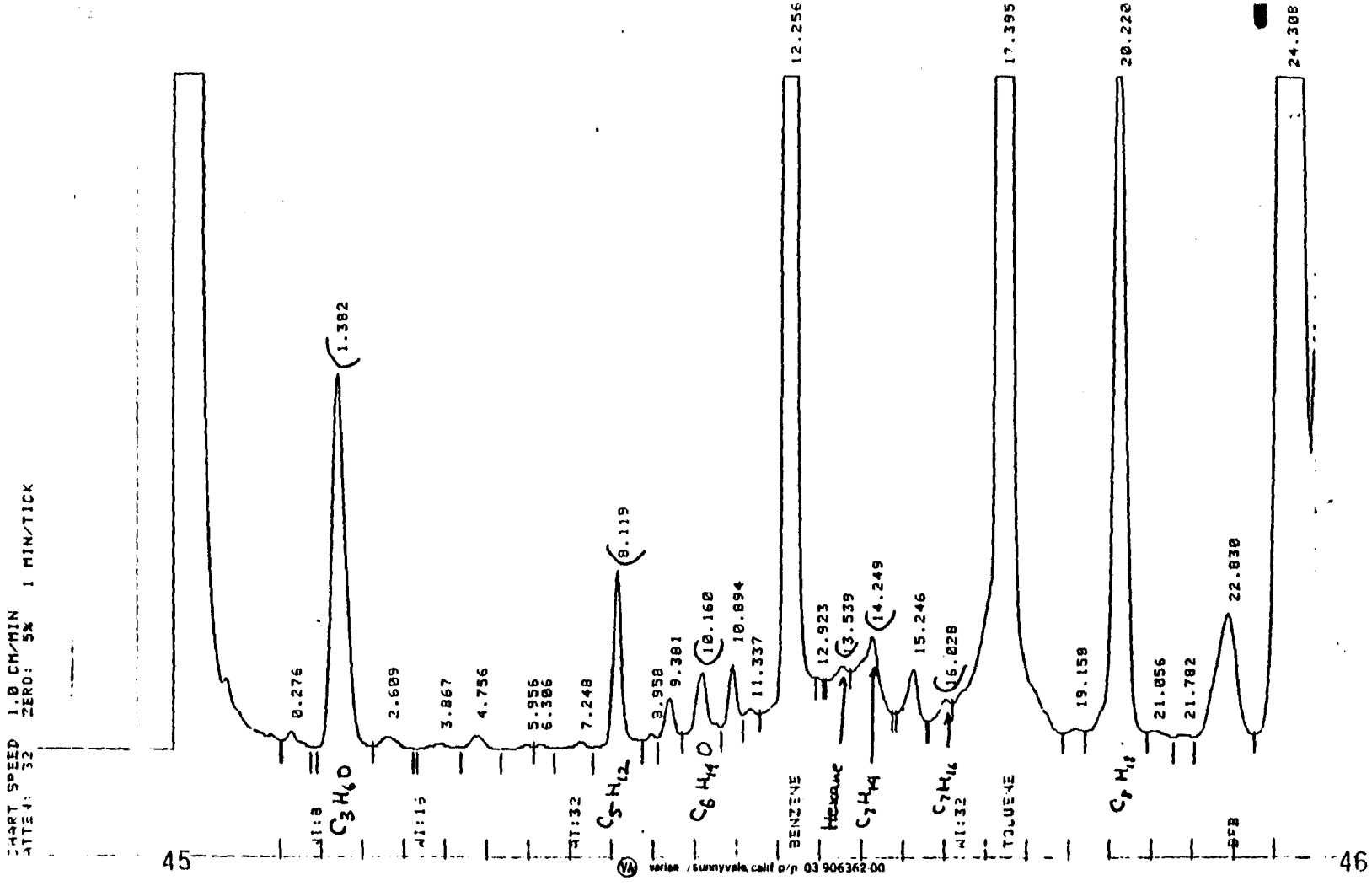


Figure 5-11. GC/FID quantitative analysis by purge and trap, sample no. IAF-INLET-VOA-0740.

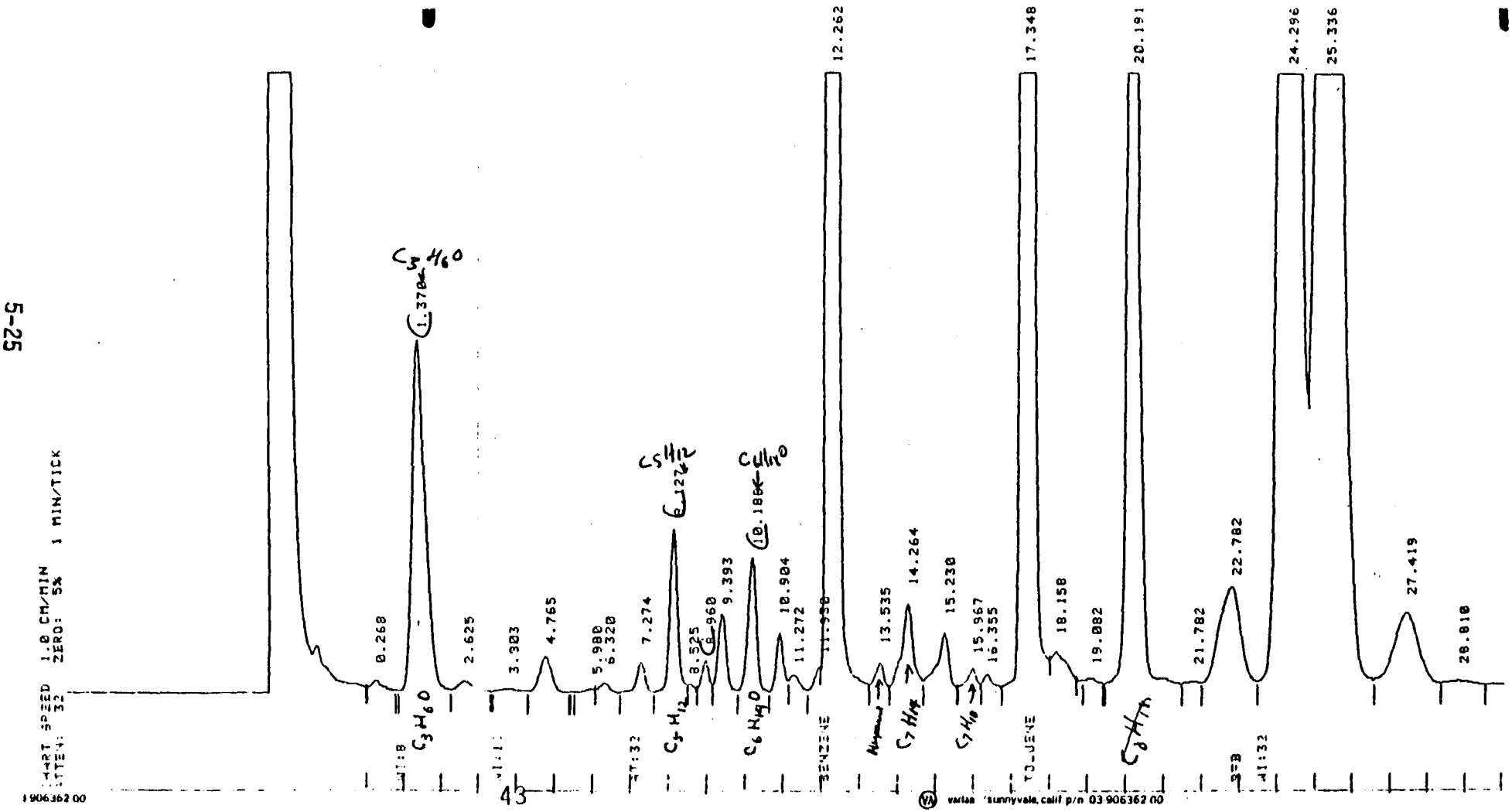


Figure 5-12. GC/FID quantitative analysis by purge and trap, sample no. IAF-OUT-VOA-0740.

Table 5-2. GC/FID READINGS FOR ACCURACY/PRECISION ESTIMATES

Compound	In-house Standard ppb	TRW Sample No. 4973					
		Replication No.					
		1 ppb	2 ppb	3 ppb	Means ppb	Std. dev. ppb	CV —
$C_2H_6S_2$	–	240	227	198	221.7	21.5	0.0970
C_6H_6	352	187	174	142	167.7	23.1	0.1381
$C_6H_5CH_3$	348	502	519	441	487.3	41.0	0.0842
BFB	596	863	927	927	905.7	37.0	0.0408

$$\% \text{ Accuracy} = \frac{905.7-596}{596} \times 100 = 51.9\%$$

% Precision = pooled CV for compounds in Sample No. 4973 = 9.6%.

Table 5-3 gives GC/FID data for an induced air flotation (IAF) sample and a dissolved air flotation sample. These samples were filtered before analysis. Accuracy estimates for IAF and DAF, respectively are 9.8 and 19.3 percent. The precision is 29.3 and 3.7 percent, respectively. In view of the fact that only duplicate analyses were performed, the precision figures for the filtered samples appears not to be significantly different (29.3 and 3.7 percent) from those for the unfiltered sample (9.6 percent). The accuracy for the filtered samples (9.8 and 19.3 percent) appear to be significantly better than the accuracy of the unfiltered sample (52 percent). It appears that the solid material in the unfiltered matrix decreased the accuracy possible in the analysis.

Table 5-3. PRECISION/ACCURACY ESTIMATES FOR IAF/DAF SAMPLES

Compound	In-house Standard	IAF, TRW #4987			DAF, TRW #4994		
		1	2	CV	1	2	CV
$C_6H_2S_2$, ppb	—	939	943	0.0030	—	—	—
C_6H_6, ppb	—	1970	1770	0.3860	2120	1980	0.0483
$C_4H_{10}S_2$, ppb	—	411	410	0.0017	—	—	—
$C_6H_6CH_3$, ppb	—	5710	5020	0.0909	2110	2000	0.0379
BFB, counts	170417	143078	164324	0.5370	135529	139579	0.0208

For IAF:

$$\text{Accuracy} = \frac{170417 - ((143078 + 164324)/2)}{170417} \times 100 = 9.8\%$$

For DAF:

$$\text{Accuracy} = \frac{170417 - ((135529 + 139579)/2)}{170417} \times 100 = 19.3\%$$

Precision:

Pooled CV for IAF = $\pm$ 29.8%.

Pooled CV for DAF = $\pm$ 3.7%.

APPENDIX A

SAMPLE CALCULATIONS AND RESULTS

- Flow and Emission Rate Calculation Examples
- Summary Gas Analysis Sheets
- Continuous Monitor Results

SUMMARY GAS ANALYSIS SHEETS

APPENDIX A

EXAMPLE CALCULATIONS

Example #1 <u>IAF - Flow Measurement with Vane Anemometers</u>

(A) $$V_{an}\ (CFM) = \frac{Ft_{an}}{min} \times Area_{an}\ Ft^2$$

(B) $$V_s\ (SCFM) = \left(\frac{V_{an} \times 17.64 \times P_b}{T + 460}\right)$$

(Example of flow measurement calculation during IAF run on 8/17/83)

$$V_{an}\ \text{Run 8-17 (CFM)} = \frac{125500\ ft}{399\ min} \times .0873\ ft^2$$

$$= 27.5\ CFM_{an}$$

$$V_s\ (SCFM) = \left(\frac{27.5 \times 17.64 \times 29.8}{117 + 460}\right)$$

$$= 25.0\ SCFM$$

SCFM = standard cubic feet per minute

V_{an} = volume measured through vane anemometer

V_s = volume standardized to standard temperature and pressure

P_b = barometric pressure

T = temperature of stack gas

Example #2 <u>Mass Emission Rate for VOC as C_3H_8</u>

(A) Sample calculation to provide the conversion factor of C_3H_8 from ppm to mg/m^3

$$CF_{C_3H_8} = \left(\frac{44\ g}{mole}\right)\left(\frac{1\ mole}{25.71\ L}\right)\left(\frac{28.32\ L}{ft^3}\right)\left(\frac{35.31\ ft^3}{m^3}\right)\left(\frac{1000\ mg}{g}\right)\left(\frac{mg/m^3}{10^6 ppm}\right)$$

$$= 1.71\ mg/m^3$$

(B) Emission rate = lb/hr

$$E_{VOC} = (\ VOC\ ppm\ )\left(\frac{1.71\ mg}{m^3}\right)\left(\frac{m^3}{35.31\ ft^3}\right)\left(\frac{Q_s\ ft^3}{min}\right)\left(\frac{60\ min}{hr}\right)\left(\frac{g}{1000}\right)\left(\frac{lb}{453.6g}\right)$$

Example - of Emission Rate calculation on IAF 8/17/83 run at 0900

$$E_{VOC} = (\ 7992\ ppm\ )\left(\frac{1.71\ mg}{m^3}\right)\left(\frac{m^3}{35.31\ ft^3}\right)\left(\frac{25.0\ ft}{min}\right)\left(\frac{60\ min}{hr}\right)\left(\frac{g}{1000\ mg}\right)\left(\frac{lb}{453.6\ g}\right)$$

$$= 1.28\ lbs/hr$$

Davis Anemometer Correction Chart

DAVIS INSTRUMENT MFG. CO., INC.
CALIBRATION CORRECTION CHART

SERIAL NO. _____

DATE June 16, 1983

TYPE BALL BEARING

TRUE F.P.M.	INDICATED F.P.M.	TRUE F.P.M.	INDICATED F.P.M.
30	18	1800	1860
50	36	2000	2065
70	53	2200	2275
90	75	2400	2490
100	85	2600	2700
200	180	2800	2910
300	290	3000	3120
400	400	3200	3335
500	505	3400	3550
600	610	3600	3765
700	715	3800	3975
800	820	4000	4180
900	925	4200	4390
1000	1030	4400	4595
1200	1235	4600	4800
1400	1445	4800	5025
1600	1655	5000	5260

WORK SHEET

R = CONC
AVERAGE
C3 15.6
C4 15.2
C5 15.6
C6 15.9

Golden West (GULR)
MINI I CALIBRATION

X ~~AFTER~~ 16
SANTA FE SPRINGS, CA

METHANE						BUTANE			$\bar{x}$	Δ	TR			
8/15	1348	1400	1435			8/15					R			
8/16	1207	1167	1192			8/16			1119	20	0.29	0.3	0.29	
8/17	1188	1148	1160			8/17			1165	20				
8/18	1218	1165	1188	1198	~~1752~~	8/18			1192	22				
8/19	1183	1123	1249	~~1551~~		8/19			1185	63				
ETHANE						PENTANE								
8/15	1519	1417	1476			8/15					0.41	0.41	.38	
8/16	1479	1362	1405			8/16			1415	59				
8/17	1404	1604	1345			8/17			1451	136				
8/18	1617	1383	1455	1514	1551	8/18			1504	?				
8/19	1576	1445	~~1275~~	1557		8/19			1526	71				
PROPANE						HEXANE					0.63	0.63	62	
8/15	2413	2077	2046			8/15								
8/16	2163	1955	2002			8/16			2042	103				
8/17	2023	2620	2167			8/17			2270	312				
8/18	2403	2058	2449	2126	2118	8/18			2231	180				
8/19	2735	2282	~~3465~~	2341		8/19			2467	238				

Aug 83

GOLDEN WEST (GWLP) Santa Fe Springs, CA

MINI CALIBRATION

WORK SHEET

									$\bar{X}$	σ	FR			
BUTANE														
8/15	2886	2670	2644											
8/16	2711	2652	2705						2689	32	1.06	1.31	1.31	
8/17	2638	3439	2792						2956	425	~~1.3~~			
8/18	2802	2738	2911	2763	2732				2789	73				
8/19	3131	3773	~~3914~~	2958					3287	429				
PENTANE														
8/15	3360	3394	3345											
8/16	3519	3443	~~4206~~						3481		~~3.169~~	3.28	3.19	
8/17	3828	3542	3436						364					
8/18	3619	3493	3731	~~4559~~	3611				3614	97				
8/19	3660	~~5352~~	3568	3910					3679	123				
HEXANE														
8/15	3715	4293	4429											
8/16	3906	4556	4281						4248	326	8.125	8.31	8.32	
8/17	4315	4617	4316						4416					
8/18	4326	~~5096~~	4102	4318	4252				4257	111				
8/19	5066	4262	4575	—					4634	405				

bmz 8-19-8

x ~~approx~~ y

WORK SHEET

o-xyl 49.9

GOLDEN WEST (GWLP) SANTA FE SPRINGS, CA
MW1 2 CALIBRATION

BENZENE									Z	A	TR			Hex	1.09
8/15	7337	7661	7802											Benz	1.29
8/16	7652	5650	8015	8190					7320	804	1.3	1.3	1.3	Hept	1.54
8/17	9179	8472	9822	8895	9746	8462	8743		9046	562				Tol	1.97
8/18	9529	~~7850~~	10354	9422	8382				9171	912				m-X	3.46
8/19	10018	8363	9755	~~11195~~	9318	9597			9110 ~~9708~~	634 ~~221~~				o-X	3.96
m-XYLENE															
8/15	7471	8392	9208												
8/16	7897	7828	7199	8280					7796	446	3.56	3.55	3.54		
8/17	~~7190~~	9956	12626	8897	12592	11064	10248		10887						
8/18	–	9413	9204	12016	11289				10486	1397					
8/19	10702	12955	10589	13905	11568	12816			11742	1145					

TRW
ENVIRONMENTAL ENGINEERING DIVISION

8/16/87

SUMMARY GAS ANALYSIS

IAF-1

COMPONENT RUN	1										
C-1	72	74.4	d6	147	144	m-x	930	935	UK	123	123
C-2	5.9	7.6	UK	448	–	o-x	322	329	UK	30.8	–
C-3	12.1	16.3	UK	229	296	UK	120	138			
C-4 UK	4.3	7.0	UK	687	670	"	615	622			
C-5 UK	25.6	31.4	UK	312	317	"	54.4	53.8			
C-6 C4	32.5	44.8	Hex	120	110	"	456	587			
BENZENE UK	151	169	Benz	1355	1359	"	129	132			
TOLUENE C5	48.8	55.2	UK	329	327	"	245	209			
UK	30.9	44.5	TOL	1347	1345	"	129	132			
XYLENE UK	11.1	72.1	UK	238	238	"	26.5	25.7			
UK	70.0	46.1	UK	218	148	"	29.3	28.5			
TOTAL HC									87.77	86.84	
%M											
% CO_2											
% CO											
% N_2	76.54		75.81								
% O_2	18.13		19.03								
% CH_4											
TOTAL %											

TRW
ENVIRONMENTAL ENGINEERING DIVISION

SUMMARY GAS ANALYSIS 8/16/83

IAF-2

COMPONENT RUN	1									
C-1	112	109	UK	1105	1102	TOL	2481	2492	UK	6.2 12.2
C-2	9.8	9.2	UK	987	928	UK	413	418		
C-3	25.1	19.2	C6	389	~~923~~ 386	UK	282	232		
C-4 UK	10	5.9	UK	222	264	H-X	1466	1451		
C-5 UK	132	125	UK	511	617	O-X	463	471		
C-6 C4	278	261	UK	1722	2078	UK	952	88		
BENZENE UK	23.2	3.8	UK	382	788		843	344		
TOLUENE UK	302	765	UK	1176	1186		302	288		
C5	256	248	HEX	374	366		189	149		
XYLENE UK	154	115	BC-NZ	2842	2860		191	186		
UK	95	—	UK	536	528		85.6	91.8		
TOTAL HC									18,968	17,167
%M										
% CO_2										
% CO										
% N_2	75.98		75.4							
% O_2	19.59		19.41							
% CH_4										
TOTAL %										

8/16/85

TRW
ENVIRONMENTAL ENGINEERING DIVISION

SUMMARY GAS ANALYSIS IAF-3

COMPONENT RUN		1								
C-1		90.8 –	UK	19 –	UK	499	485	290 ~~107.4~~	280
C-2		9.6 –	UK	730 –	TOL	2322	2320	162 ~~7.3~~	155
C-3		14.4 –	UK	699 –	UK	450	452	16.4	13.8
C-4	UK	7 –	C6	256 –	UK	325	325	7.3	44.4
C-5	UK	53.9 –	UK	20 –	M-X	1578	1578		
C-6	C4	108 –	UK	255 –	O-X	511	508		
BENZENE	UK	2.8 –	UK	1735 –	UK	180	186		
TOLUENE	UK	402 –	UK	678 –	UK	928	107		
	C5	130 –	UK	548	533	UK	1067	1061	
XYLENE	UK	61.9 –	HEX	1063	1073	UK	~~430~~ 433	424	
	UK	8.5 –	BENZ	2424	2425	UK	~~282~~ 276	283	
TOTAL HC							MINUS 1 + MINUS 2 = TTL	5467+ 12244 17,711	12268 17,735
%M									
% CO_2		0.93		0.97					
% CO									
% N_2		74.38 ~~5.90~~		72.55					
% O_2		18.66 ~~5.94~~		18.38					
% CH_4									
TOTAL %									

TRW
ENVIRONMENTAL ENGINEERING DIVISION

8-12-83

SUMMARY GAS ANALYSIS

IAF-1

CONTINUED

COMPONENT RUN	1									
C-1	134	141	UK	42.4	31.4	benz	1769	1748	UK	254 243
C-2	7.6	8.0	UK	609	639	UK	408	398	UK	180 164
C-3	19.6	18.4	UK	596	534	TOL	1635	1623	UK	34 30
C-4 UK	8.7	6.7	C4	335	316	UK	324	321	UK	109 104
C-5 UK	61.5	59.0	UK	1.2	–	UK	296	285	UK	17.4 27.2
C-6 C4	141	140	UK	173	114	m-xyl	909	900		
BENZENE UK	2.8	4.1	UK	447	383	o-xyl	312	298		
TOLUENE UK	404	392	UK	1420	1313	UK	163	157		
C5	186	179	UK	449	446	UK	80.4	–		
XYLENE UK	80.4	~~74~~ 71.6	UK	620	618	UK	63.5	57.5		
UK	6.6	–	HEX	180	179	UK	483	120		
TOTAL HC									12,972 ~~13,272~~	12,074
%M										
% CO_2										
% CO										
% N_2	76.56		75.29							
% O_2	21.84		21.48							
% CH_4										
TOTAL %										

TRW

ENVIRONMENTAL ENGINEERING DIVISION

8-12-83

SUMMARY GAS ANALYSIS

IAF-2

COMPONENT RUN													
		ppm											
C-1		126	145	UK	35.2	88.6	HEX	1151	1160	UK	222	222
C-2		17.7	34.2	UK	170	-	BEN2	7293	7259	UK	337	330
C-3	UK	1.6	-	UK	2542	2520	UK	2555	2557	UK	264	270
C-4	C3	78.1	79.0	UK	2928	2860	TOL	4776	4728	UK	41.6	-
C-5	UK	34.8	35.3	C6	1576	1605	UK	1418	1392	UK	84.5	113
C-6	UK	139	139	UK	521	563	UK	1173	1154	UK	170	168
BENZENE	C4	316	313	UK	2971	2972	M-XY	1624	1628	UK	89.2	98.3
TOLUENE	UK	48.9	40.7	UK	6084	6293	O-XY	573	562	UK	15.2	11.1
	UK	1709	1700	UK	2060	2250	UK	437	433			
XYLENE	C5	695	676	UK	2326	2318	UK	211	206			
	UK	298	298	UK	2765	2710	UK	142	142			
TOTAL	HC										50,031	50,070
%M												
% CO_2												
% CO												
% N_2		73.68		73.82								
% O_2		20.04		20.03								
% CH_4												
TOTAL %												

TRW
ENVIRONMENTAL ENGINEERING DIVISION

8-17-83

SUMMARY GAS ANALYSIS

IAF-3

COMPONENT RUN		1	~~2~~	1	2	1	2	1	2
C-1		262	–	UK	2784 –	UK	2529	2528	UK 357 372
C-2	C3	122	–	UK	~~1669~~ 2725 –	TOL	4912	4995	UK 309 313
C-3	UK	48.7	–	C6	1669 –	UK	1424	1462	UK 141 106
C-4	UK	179	–	UK	616 –	UK	1216	1237	UK 183 184
C-5	C4	365	–	UK	3008 –	M-XYL	1761	1779	UK 113 112
C-6	UK	162	–	UK	6251 –	O-XYL	606	627	UK 13.3 12.4
BENZENE	UK	1848	–	UK	2284 –	UK	488	505	
TOLUENE	UK	1218	–	UK	2176 2215	UK	228	235	
	C5	341	–	UK	2688 2650	UK	145	165	
XYLENE	UK	279	–	HEX	1020 1076	UK	817	822	
	UK	183	–	BENZ	7016 7104	UK	244	256	
TOTAL HC									
%M									
% CO_2		0.41		0.4					
% CO									
% N_2		73.19		72.99					
% O_2		21.16		21.09					
% CH_4									
TOTAL %									

	run 1	run 2
min. 1	24,345	–
min. 2	28,386	28,755
	52,731	

TRW

ENVIRONMENTAL ENGINEERING DIVISION

8-18-83

SUMMARY GAS ANALYSIS

IAF-2

COMPONENT RUN		1						
C-1		94.7	UK	2.4	TOL	1661	UK	206
C-2		4.1	UK	15.7	UK	200	UK	41.7
C-3		8.0	UK	529	UK	186	UK	116
C-4	UK	3.0	C6	149	M-XYL	1164		
C-5	UK	37.7	UK	2.3	O-XYL	407		
C-6	C4	96.5	UK	42.6	UK	151		
BENZENE	UK	1.5	UK	107	UK	79.3		
TOLUENE	UK	1.6	UK	238	UK	59.7		
	UK	196	HEX	81.4	UK	520		
XYLENE	C5	71.0	benz	1106	UK	132		
	UK	30.4	UK	224	UK	277		
TOTAL	HC						8243	
%M								
% CO_2		1.18	1.18					
% CO								
% N_2		72.[illegible]	73.28					
% O_2		21.19	21.23					
% CH_4								
TOTAL %								

TRW
ENVIRONMENTAL ENGINEERING DIVISION

8-18-83

SUMMARY GAS ANALYSIS

IAF-1

COMPONENT RUN	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	
	1						
C-1	44.5	–	UK	201 –	benz	552 542	UK 116 103
C-2	3.0	–	C6	60.1 –	UK	138 137	UK 243 226
C-3	4.2	–	UK	7.1 –	TOL	895 883	UK 161 152
C-4 UK	2.0	–	UK	1.6 –	UK	154 141	UK 20.7 13.6
C-5 UK	8.2	–	UK	6.1 –	UK	150 143	UK 81.9 86.1
C-6 C4	10.5	–	UK	4.1 –	H-XYL	657 638	
BENZENE UK	1.6	–	UK	92.4 –	P-XYL	242 230	
TOLUENE UK	34.2	–	UK	38.2 34.0	UK	125 116	
C5	14.9	–	UK	27.8 23.7	UK	56.7 63.2	
XYLENE UK	10.0	–	C6	49.3 50.	UK	50.6 53.6	
UK	5.5	–	UK	34.8 33.4	UK	413 390	
TOTAL HC							
%M							
% CO_2	0.84		0.72				
% CO	~~*~~						
% N_2	76.89		74.59				
% O_2	21.93		21.21				
% CH_4							
TOTAL %							

	Run 1	Run 2
MIN. 1	511.	–
MIN. 2	~~5177~~	
TOTAL	4206	4055
	~~5228~~	
	4717	

TRW

ENVIRONMENTAL ENGINEERING DIVISION

8-19-87

SUMMARY GAS ANALYSIS

IAF-1

COMPONENT RUN	1									
C-1		64.3	67.8	UK	48	–	UK	217	216	UK	190	189
C-2		6.6	4.1	UK	18.	10.1	TOL	1112	1103	UK	97.3	95.8
C-3		8.8	7.4	UK	530	527	UK	187	180	UK	11.1	4.
C-4	UK	8.6	7.2	C6	153	151	UK	166	165	UK	39.2	15.6
C-5	UK	16.4	14.7	UK	24.9	12.3	M-XYL	640	640			
C-6	C4	28.9	27.9	UK	14.5	23.	O-XYL	223	234			
BENZENE	UK	6.5	11.8	UK	34.4	–	UK	104	103			
TOLUENE	UK	1.	–	UK	160	160	UK	52.6	214			
	UK	137.4	170.3	UK	235	242	UK	41	34.8			
XYLENE	C5	80.4	100.3	HEX	97.3	85.7	UK	343	343			
	UK	41.1	73.3	BENZ	871	860	UK	88.4	87.7			
TOTAL HC											6054	6194
%M												
% CO_2		0.48			0.38							
% CO												
% N_2		73.09			74.13							
% O_2		23.58			23.28							
% CH_4												
TOTAL %												

TRW
ENVIRONMENTAL ENGINEERING DIVISION

8-18-83

SUMMARY GAS ANALYSIS

IAF-2

COMPONENT RUN	1							
C-1	71.7	74.0	UK	1.3	–	UK	274 280	UK	195 199
C-2	6.0	7.6	UK	42.9	12.0	TOL	1774 1797	UK	97.2 98.1
C-3	9.9	11.9	UK	738	667	UK	222 220	UK	2.8 –
C-4 UK	6.3	12.6	C6	192	187	UK	196 195		
C-5 UK	22.3	27.5	UK	36.5	21.8	M-XYL	882 898		
C-6 C4	47.4	54.0	UK	–	23.2	O-XYL	295 298		
BENZENE UK	4.5	3.5	UK	32.2	76.5	UK	115 119		
TOLUENE UK	168	178	UK	153	182	UK	55.8 57.6		
C5	77.7	80.2	UK	290	295	UK	42.8 44.2		
XYLENE UK	41.1	39	UK	116	117	UK	384 383		
UK	8.5	–	BENZ	1232	1240	UK	90.3 88.7		
TOTAL HC								7953	7987
%M									
% CO_2	0.34		0.33						
% CO									
% N_2	75.27		27 64.39		68.04				
% O_2	28.17		27.61		27.44				
% CH_4									
TOTAL %									

CONTINUOUS MONITOR RESULTS

TRW

LOCATION Golan West IAF Quarry POLLUTANT VOC DATE 8/15/83 → 8/16/83

INSTRUMENT RANGE (PPM) 0 – 10,000 ppm CALIBRATED BY GBS

Record Data Every 3-5 Minutes 4010 ppm CH_3 SPAN

Time	Avg Scale Reading	Avg ppm/HR
1600	70.92	7092
1700	70.42	7042
1800	69.25	6925
1900	67.92	6792
2000	64.0	6400
2100	62.58	6258
(A) 2400	60.50	6050
0100	62.50	6250
0200	61.25	6125
0300	61.0	6100
0400	63.0	6300
0500	67.5	6750
0600	66.75	6675
0700	68.44	6844
0800	67.00	6700
0900	64.58	6458
1000	67.25	6725

Time	Scale Reading	ppm
1100	74.83	7483
1200	72.67	7267
1300	69.08	6908
1400	~~47.17~~ 62.83	~~4717~~ 6283
1500	58.50	5850
1600	58.50	5850
1700	60.58	6058
1800	57.25	5725
1900	56.50	5650
2000	61.0	6100
2100	62.33	6233
2200	63.0	6300
2300	67.08	6708
2400	68.83	6883

NOTES: (A) 2200 61.7 6170
2300 60.83 6083

TRW

LOCATION Edwards West IAF Outlet POLLUTANT VOC DATE 8/17/83

INSTRUMENT RANGE (PPM) 0-10000 ppm CALIBRATED BY CBS

Record Data Every 3-5 Minutes 4010 ppm CH_3 SPAN

Time	Avg ~~Scale Reading~~	Avg ppm/hr
0100	69.25	6925
0200	67.83	6783
0300	60.75	6075
0400	66.08	6608
0500	69.50	6950
0600	70.33	7033
0700	71.50	7150
0800	68.67	6867
0900	79.92	7992
1000	86.75	8675
1100	85.92	8592
1200	88.11	8811
1300	75.0	7500
1400	65.08	6508
1500	65.58	6558
1600	61.75	6175
1700	63.67	6367

Time	Scale Reading	ppm
1800	66.67	6667
1900	69.58	6958
2000	73.08	7308
2100	73.92	7392
2200	70.17	7017
2300	68.25	6825
2400	69.17	6917

NOTES:

TRW

LOCATION Golden West IAF Outlet POLLUTANT VOC DATE 8/18/83

INSTRUMENT RANGE (PPM) 0 - 10,000 CALIBRATED BY GBS

Record Data Every 3-5 Minutes 4010 ppm CH_3 Span

Time	Scale Reading	ppm
0000	69.17	6917
~~0100~~ 0100	71.92	7192
0200	73.00	7300
0300	74.83	7483
0400	75.00	7500
0500	74.73	7473
0600	69.42	6942
0700	68.00	6800

Time	Scale Reading	ppm

NOTES: * Overlap from previous day

TRW

LOCATION IAF Outlet POLLUTANT VOC DATE 8/18/83

INSTRUMENT RANGE (PPM) 0 - 10,000 CALIBRATED BY CBS

Record Data Every 3-5 Minutes 4010 ppm HC_3 SPAN

	Time	Scale Reading	ppm
	1000	60.10	6010
	1100	59.40	5940
	1200	64.75	64.75
	1300	66.50	6650
1430 1451	1400	68.00	6800
Calibrate	1500	71.50	71.50
	1600	70.50	7050
	1700	67.50	6750
	1800	67.00	6700
	1900	64.40	6440
	2000	67.10	6710
	2100	66.50	6650
	2200	66.30	6630
	2300	67.50	6750
	2400	66.60	6660
	0100	64.40	6440
	0200	62.10	6210

	Time	Scale Reading	ppm
	0300	59.20	5920
	0400	59.00	5900
	0500	60.90	6090
	0600	61.50	6150
	0700	63.60	6360
0815-0824 Cal.	0800	62.00	6200
	0900	62.10	6210
	1000	63.25	6325
	1100	66.30	6630
	1200	67.60	6760
	1300	66.40	6640
	1400	OFF	Line

NOTES:

APPENDIX B

FIELD DATA SHEETS

IAF Anemometer Measurements

TRW

IAF Anemometer Flow Measurements

LOCATION Baldwin West POLLUTANT _____ DATE 8/19/83

INSTRUMENT RANGE (PPM) _____ CALIBRATED BY _____

Record Data Every 3-5 Minutes

Time	~~Scale~~ Reading	Temp °F ~~ppm~~
08:34:00	0000	115
08:46:25	5000	
09:09:24	15000	
09:54:34	35000	
10:34:30	52300	
10:54:10	61000	
11:35:00	80000	
12:00	90000	
12:53:05	104000	
13:00:00	115300	
13:34:38	130000	
14:03:05	142000	

Time	Scale Reading	ppm

NOTES:

Anemometer

~~PRELIMINARY VELOCITY~~ TRAVERSE

PLANT Golden West
DATE 8/18/83
LOCATION ~~IQF~~
STACK I.D.
BAROMETRIC PRESSURE, in. Hg
STACK GAUGE PRESSURE, in. H_2O
OPERATORS D. B. Savia

Reading Time

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
~~0000~~	~~0800~~	~~76~~
0000	10:26:00	111
15000	11:10:45	110
25000	11:39:25	110
53500	12:59:00	118
71460	13:51:00	120
77400	14:07:48	120
86600	14:32:19	119
98000	15:02:47	119
AVERAGE		

SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F

PRELIMINARY ~~VELOCITY TRAVERSE~~ Anemometer

PLANT Golden West
DATE 8/17/83
LOCATION IAF
STACK I.D. _____
BAROMETRIC PRESSURE, in. Hg _____
STACK GAUGE PRESSURE, in. H_2O _____
OPERATORS D. Savin

Reading Time

~~TRAVERSE~~ POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
0000	0740	115°
4900	0750	115
10000	0808:00	115
20300	08:36:31	117
40000	09:40:33	118
50000	09:55:35	100
64300	10:39:22	100
69000	10:53:56	120
85000	11:44:15	119
90000	12:00:11	119
102000	12:41:20	118
104100	12:49:57	118
108500	13:08:03	119
113600	13:29:09	117
117800	13:46:44	115
125500	14:19:13	114
		117
	6:39 -	
		314 fpm
AVERAGE		

SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F

~~PRELIMINARY~~ VELOCITY ~~TRAVERSE~~ (Anemometer)

PLANT Golden West
DATE 8/16/83
LOCATION IAF - OUT
STACK I.D.
BAROMETRIC PRESSURE, in. Hg
STACK GAUGE PRESSURE, in. H_2O
OPERATORS David Savin

Reading Elapsed Time

TRAVERSE POINT NUMBER	VELOCITY ~~HEAD~~ (Δp_s), ft/sec	STACK TEMPERATURE (T_s), °F	
2630	7 07:17	122	
3700	7 10:15	122	
5000	7 14:05	123	
6500	7 17:55	122	
7600	7 21:00	122	
11600	7 31:55	122	
13400	7 36:50	123	
16500	7 42:28	123	
19300	7 52:35	123	
31800	8:25:24	123	
37700	8:28:00	123	0911
38400	8:25:39	123	
41400	8:33:28	123	
48300	8:51:23	124	
60000	10:21:44	124	
64500	10:33:22	124	
83700	11:24:21	124	
97000	11:59:36	126	
110300	12:08:25	126	Stop
	4.19 hrs		
		110300	= 125 fpm
AVERAGE			

2.22

SCHEMATIC OF TRAVERSE POINT LAYOUT

TRAVERSE POINT NUMBER	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
0000	12:28:00	127
1470	12:32:00	127
8700	12:54:07	128
19200	13:20:00	128
35500	13:47:15	129
46900	14:12:57	129

Liquid Sampling Log

WORK SHEET

Sampling Logo

Golden West Refining Co.

8/16/83

~~Pg~~ 2

	Day		8/16											~~8/18~~
①	Bag Sample	Start	7:35	~~Stop 8:35~~					0745	1000	1153			
		Stop	8:35						0845	1100	1253			
	Von Samples													
	IAF-IN		8:05						0740			1330		✓
	IAF-out		8:10						0740			1330		
	Composite Samples													
	IAF-IN	START	8:05						0740	10	11	1300		
	~~IAF-out~~	STOP												
	IAF-out	START	8:10	905	1000	1200	1300		0740	10	11	1300		
		STOP												
	API-IN	START	8:30						0740	10	11	1100		
		STOP												
②	Bag Sample	START	10:20											
		STOP												

Q

WORK SHEET

Sampling Log — Golden West Refining Co. 2 of 2

Day		8/18				8/19								
BAC Samples	START	1030	1310		START	850 ①	1030							
	STOP	1146	1410		STOP	950	1130							
VOA Samples														
IAF - IN		1040	1510			830								
IAF - OUT		1040	1510			830								
Composite Samples		START	STOP			START	STOP							
IAF - IN		1040	1510			830	1400							
IAF - OUT		1040	1510			830	1400							
API - IN	START	1136			START	830	~~1400~~							

① W. Kelly + GDH performed QUA Monitoring on DRAINS

APPENDIX C

ANALYTICAL DATA

- Gas Chromatograph Worksheet
- Continuous Monitor Example
- GC/FID Examples of IAF #3 Analysis on 8/16/83

GC WORKSHEETS

GC WORKSHEET

COLUMN: 735-835 AM RUN NUMBER: IAR-1 DATE: 8/16/83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN FS	ATTENUATION CTS	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE R
C_1	.29	5333	5662	5662		72 76.4	0.0135
C_2	.4	568	684	684		5.9 7.0	0.0103
C_3	0.63	1582	2135	2135		12.1 16.3	0.0076
C_4 WK	0.86	565	924	924		4.3 7.0	as propane
UK	1.11	3354	4106	4106		25.6 31.4	"
C_5 C4	1.31	5755	7434	7434		32.5 44.8	0.0057
C_6 UK	2.71	1974	22185	22185		151 169	0.0076
C5 ~~UK~~	3.17	10882	12330	12330		48.8 55.2	0.0045
UK BENZENE	3.57 5.31	4046	5824	5824		30.9 44.5	0.0076
		1456	~~[illegible]~~	~~[illegible]~~ 9431		11.1 72.1	0.0076
TOLUENE UK	UK 7.46 7.36	41559	~~[illegible]~~	60349		700 461	
C4	8.32 8.16	39181	~~[illegible]~~	38551		147 144	0.0037
XYLENE C4	13.79	5858 ~~5553~~	~~[illegible]~~			44.8 –	0.0076
UK	15.46 15.23	30005	~~[illegible]~~	38771		229 294	
UK	18.19 17.9	8997	~~[illegible]~~	82640		687 670	
TOTAL HYDRO-CARBONS (THC)							

Golden West (Gulf) MIN. 2

GC WORKSHEET

COLUMN: _____ RUN NUMBER: IAF-1 DATE: 8/16/83

COMPOUND	RETENTION TIME IN CM.	COUNTS	~~SPAN~~ CTS	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1 unk	1.05	45872	46650			312 317	as benzene 0.0068
C_2 hex	1.15	17722	16211			120 110	
C_3 benz	1.32	199119	199782			1355 1359	
C_4 unk	1.7	48208	48084			328 327	
C_5 TOL	1.88	198409	197710			1347 1345	
C_6 unk unk	2.45 3.- 3203	35049 ~~35049~~	35000 31689			238 238 218 148	
m-xyl o-xyl BENZENE	3.47 3.45 145284	~~32073~~ 50340	146036 51447			430 435 322 329	0.0064 0.0064
TOLUENE	4.55 4.45 4.95 4.85	17634 9139	18789 9194			120 128 61.5 62.2	as benzene
XYLENE	5.41 5.29 6.27 6.13	3073 67082	7904 86293			54.9 53.8 456 587	
	6.83 7.73 7.56 9.1 8.9	19014 36064 18919	19430 37062 19430			129 132 245 209 129 132	
	9.98 9.8 10.81 10.5	3897 4312	3775 4253			26.5 25.7 29.3 28.5	
TOTAL HYDRO-CARBONS (THC)	11.85 14.1	18147 4524	18009			123 123 30.8	

TRW
ENVIRONMENTAL ENGINEERING DIVISION

M 10 11.

GC WORKSHEET

COLUMN: 1020-~~1030~~ 1130 RUN NUMBER: 7A-F-2 DATE: 8/6/83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN CTS	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1	0.3	8269	8112			112 109	0.0135
C_2	0.4	949	891			9.8 9.2	0.0103
C_3	1.61	3290	2518			25.4 19.2	0.0076
C_4 ~~WK~~ ~~WK~~	0.81 1.05	1306 17285	776 16410			10 5.9 132 125	0.0076 as " propane
C_5 C4	1.22	49097	46132			278 261	0.0057
C_5 ~~WK~~ ~~WK~~	1.81 1.75 2.47 2.38	3041 104984	504 100193			23.2 3.8 862 765	as propane " 0.0076
C5 BENZENE	2.87 2.76	57180	54527			256 244	0.0045
WL TOLUENE	3.26 3.11 4.76 ~~4.71~~	20115 12434	15027			154 115 95 -	as propane 0.0076
XYLENE	6.09 5.78 6.59 6.31	144584 129218	144187 121755			1105 1102 987 928	
C6 →	~~2.00~~ C6 6.51 11.86 11.28 13.46 12.75	103423 29000 66886	103265 34511 80712			387 386 222 264 511 612	0.0037 as propane
	15.52 14.75 18.72 17.68	225456 50009	271978 103131			1722 2078 382 788	
TOTAL HYDRO-CARBONS (THC)							

m1W12

GC WORKSHEET

COLUMN: 1020-1130 RUN NUMBER: IAP-2 DATE: 8/16/87

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1 unk	1.03	172798	174340			1176 1186	as benzene 0.0068
C_2 Alt	1.1	54967	53761			374 366	
C_3 Benz	1.28	417719	420421			2842 2860	
C_4 unk	1.63	78766	77541			536 528	
C_5 TOL	1.87	364750	366266			2481 2492	
C_6 unk C_6 unk	2.27 2.78	61462 41471	61481 41482			418 418 282 282	
m-xyl BENZENE	3.2 3.18	229001	226650			1466 1451	0.0064
o-xyl TOLUENE	3.65 3.61	22353	23639			463 471	0.0064
unk XYLENE	4.13 4.09 4.61 4.45	21347 13589	21582 12933			146 147 95.2 88	as benzene
	5.67 5.62 6.97 6.87 8.13 8.04	124578 44046 27838	124137 42420 21958			848 844 300 288 189 149	
	10.47 10.33 12.61 12.37	28143 12576	27895 13498			191 186 85.6 91.8	
TOTAL HYDRO-CARBONS (THC)	14.24 14.08	908	1795			6.2 12.2	

GC WORKSHEET

COLUMN: 1230-130 RUN NUMBER: IAF-3 DATE: 8/16/87

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1	0.3	6734				90.8	0.0135
C_2	0.39	931				9.6	0.0103
C_3	0.6	1.887				14.4	0.0076
C_4 UK UK	0.77 1.	913 7051				~~108~~ 7 53.9	as propane 0.0076
C_5 C4	1.15	19022				108	0.0057
C_5 UK UK	1.67 2.3	300 52614				2.8 402	as propane 0.0076
C5 BENZENE	2.65	29131				130.	0.0045
TOLUENE UK UK	3. 3.98	8659 1117				61.9 8.5	as propane 0.0076
XYLENE UK UK	4.33 5.51	2983 95587				19 730	
UK C6	5.98 6.51	91525 68406				699 256	0.0037
UK ~~TOTAL~~ UK ~~TOTAL~~	10.61 12.01	26700 68118				266 255	as propane 0.0076
TOTAL HYDRO-CARBONS (THC)	13.75 16.41	227161 83746				173.5 ~~66~~ 67.8	

MINI 2

GC WORKSHEET

COLUMN: 1230-130 RUN NUMBER: IAF-3 DATE: 8/16/83

COMPOUND	RETENTION TIME IN CM.	COUNTS	~~SPAN~~ cps	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND ppm	CONCENTRATION AS ~~BENZENE~~ R
C_1 UK ~~UK~~	0.82	80538	79086			548 538	as benzene 0.0068
C_2 hex	1.62	156277	157723			1063 1073	
C_3 benz	1.25	356181	356480			2424 2425	
C_4 UK	1.57	73390	72841			499 495	
C_5 Tol	1.8	341358	340953			2322 2320	
C_6 UK UK	2.15 2.62	66129 47801	64560 47718			450 452 ~~504~~ 325 325	
BENZENE	3.01 3.64	246564 79865	246567 79458			1578 1578 511 508	0.0064 0.0064
TOLUENE	3.83 4.18	26431 13640	27386 19070			180 186 92.8 102	as benzene
XYLENE	5.23 6.39	158822 63208	158846 63052			1067 1061 433 429	
	7.43 9.49 11.32	40513 42567 23872	41590 41166 22825			276 283 290 280 162 155	
	13.11 14.65	2408 1079	2037 6530			16.4 13.8 7.3 44.4	
TOTAL HYDRO-CARBONS (THC)							

m-xyl
o-xyl
UK

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MIN 16

GC WORKSHEET

COLUMN: 0730-0830 RUN NUMBER: IAF-1 DATE: ~~8/17~~ 8/17/83

COMPOUND	RETENTION TIME IN CM.		COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND		CONCENTRATION AS ~~BENZENE~~ R
C_1	.29		7625	7988			134	141	0.0176
C_2	.34		751	793			7.6	8.0	0.0101
C_3	.63	.62	2852	2673			19.6	18.4	0.0069
C_4 UK	.88	.84	1272	876			8.7	6.7	as propane
UK	1.12	1.07	8945	8585			61.5	59.0	"
C_5 C4	1.32	1.26	27334	27155			141	140	" 0.0051
C_6 UK	2.08	1.91	404	597			~~1.83~~ 2.8	4.1	as propane
UK	2.74	2.53	58804	57096			404	392	"
C5 BENZENE	3.23	3.04	42878	41283			186	179	0.0043
TOLUENE UK	3.64	3.41	11702	10423			80.4	71.6	as propane
	4.24		957				6.6	–	"
XYLENE UK	5.44	5.03	6167	4565			42.4	31.4	"
UK	7.03	6.46	38650	42963			609	639	"
UK	7.58	7.03	36724	27749			596	534	"
C6	8.51	7.76	43030	37840			335	316	0.0036
UK	11.64	12	164				1.1	–	as propane
	14.11	12.76	25844	16545			178	114	"
	15.81	14.3	65065	56396			447	388	"
TOTAL HYDRO-CARBONS (THC)	18.14	16.7	206584	191072			1420	1313	"

.08
.60
430

MIXI 2

GC WORKSHEET

COLUMN: 0730-0830 RUN NUMBER: IAS-7 DATE: 8-17-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1 uk	.85	81569	81040			449 446	co benzene 0.0055
C_2 uk ~~hex~~	1.04	112640	112183			620 618	"
C_3 hex	1.12	32657	32460			180 179	"
C_4 benz	1.32	321354	317539			1769 1748	"
C_5 uk	1.7	74042	72357			408 398	"
C_6 TOL	1.98	297004	294827			1635 1623	"
BENZENE uk	2.47	58858	58362			324 321	"
~~[illegible]~~ m-xyl o-xyl TOLUENE	3.03 53934 3.52 19836	~~[illegible]~~ ~~6858~~	52592 16420		296 → ~~909~~	~~[illegible]~~ 289 909 900	~~[illegible]~~ " 0.0016
XYLENE	4.1 68150 4.6 29637	~~[illegible]~~ ~~14645~~	64983 28477			312 298 163 157	~~[illegible]~~ 0.0046 ~~co benzene~~
	5.02 14648 6.38 5.5 11519 7.09 8873 24213	~~[illegible]~~ ~~[illegible]~~ ~~24213~~	— 10446 87995 21716			80.6 — 63.4 57.5 488 120	
	7.83 116221 ~~[illegible]~~ 9.3 32744	~~46221~~ ~~32744~~	44085 29767			254 243 180 164	
TOTAL HYDRO-CARBONS (THC)	11.05 6174 12.01 19356 14.46 3163	~~6174~~ ~~19356~~ ~~3163~~	~~5489~~ 18945 4936	5489		34 30 109 104 17.4 27.2	

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MINI 1

GC WORKSHEET

COLUMN: 1020 -1120 RUN NUMBER: 1 AF-2 DATE: 8-17-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1	0.3	7173	8214			126 145	0.0176
C_2	0.39	1759	2407			17.7 24.2	0.0101
~~C_3~~ UK	0.5	232	–			1.6 –	as propane 0.0069
~~C_4~~ C3	0.6	11365	11499			78.1 79.0	0.0069
C_5 UK2 UL	0.8 1.03	5042 20261	5137 20134			34.8 35.3 139 139	" "
C_6 C4	1.2	61457	60788			316 313	0.0051
BENZENE UK UK4	1.79 2.43 2.35	7119 248723	5924 247402			48.9 40.7 1709 1700	as propane "
C6 TOLUENE	2.81 2.75	160348	156038			695 676	0.0043
XYLENE UL	3.18 3.56	43389 5122	43312 12898			298 298 35.2 88.6	as propane "
	4.3 – 5.83 6.5 6.41	24716 369840 426055	– 366603 416156			170 – 2542 2520 2928 2860	" " "
C6 C4	7.2 11.66	437603 75800	445793 81987			1576 1605 521 563	0.0036 as propane
TOTAL HYDRO-CARBONS (THC)	13.13 15.73 15.44 18.26 17.91	432277 985276 294763	432439 915664 327350			2971 2972 6084 6293 2060 2250	" " "

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MINI 2

GC WORKSHEET

COLUMN: 1020-1120 RUN NUMBER: 3AF2 DATE: 8-17-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1 UIL	0.88	4122492	4210887			2326 2318	as benzene 0.0055
C_2 UIL	1.03	5032286	4933565			2765 2717	"
C_3 hex	1.1	209084	210672			1151 1160	"
C_4 benz	1.29	1324784	1318561			7293 7259	"
C_5 TOL	1.61 1.86	464104 867484	464473 858792			2555 2557 4776 4728	"
C_6	2.26 2.75	257672 217134	252813 209645			1418 1392 1173 1154	" "
m-+41 o-+41 BENZENE	3.15 3.58	354250 124935	355104 122725			1624 1628 573 562	0.0046 0.0046
TOLUENE	4.07 4.45	79784 38282	78677 37461			439 433 211 206	as benzene "
XYLENE	4.84 5.54 5.48	25703 136062	25774 133608			142 142 749 736	" "
	6.14 6.06 6.8 6.72 7.9 7.73	40344 61255 48042	40305 59868 48987			222 222 337 330 264 270	" " "
	8.8 9.32 9.16	7559 15353	— 20528			41.6 — 84.5 113.	" "
TOTAL HYDRO-CARBONS (THC)	10.24 12.25 14.2	36848 18026 2767	30440 17862 2018			170 168 99.2 98.3 15.2 11.1	" " "

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MINI

GC WORKSHEET

COLUMN: 1153-1253 RUN NUMBER: IAP-3 DATE: 8-17-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1	0.29	14900				262	0.0176
~~C_2~~ C3	0.61	12155				122	0.0069
~~C_3~~ 4K 4K	0.79 1.02	7085 25997				48.7 179	as propane 0.0069 "
C_4 C4	1.19	71046				365	0.0051
C_5 4K 4K	1.76 2.37	23594 268853				162 1848	as propane "
C_5 ~~C5~~ 4K	2.76 177256	~~78637~~				1218	"
C5 ~~BENZENE~~ 4K	3.12 78682 ~~193696~~ 4.19 40547	40547 ~~36649~~				341 279	0.0043 as propane
TOLUENE 4K 4K	4.54 5.79	26649 405843				183 2784	" "
XYLENE 4K C6	6.32 7.02	356561 463605				2725 1669	" 0.0036
	11.36 12.83 15.42	85651 437724 909640				616 3003 6251	as propane " "
	17.82	332310				2284	"
TOTAL HYDRO-CARBONS (THC)							

.06/360

MIW 2

GC WORKSHEET

COLUMN: 1153-1253 RUN NUMBER: 145-3 DATE: 8-12-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN 45	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND		CONCENTRATION AS ~~BENZENE~~ R
C_1 unk	.88	395204	402437			2176	2215	as benzene " 0.0055
C_2 unk	1.62	488189	481295			2638	2650	"
C_3 hex	1.08	185387	195442			1020	1076	"
C_4 benz	1.27	1274446	1290482			7016	7104	"
C_5 tol	1.57 1.81	459446 892367	459155 907564		 4912	2529 ~~2529~~	2523 4995	" "
C_6	2.19 ~~2.49~~ 2.67	268588 220940	265584 224675		 1216	1421 ~~1423~~	1462 1237	" "
BENZENE	3.05 3.47	384237 132303	392532 136862			1761 606	1794 627	0.0046 0.0046
TOLUENE	3.91 4.29	88633 41385	91690 42699			488 228	505 235	as benzene "
XYLENE	4.66 5.32	26417 148426	29848 149300			145 817	165 822	" "
	5.88 C. 6.52 7.54	44305 64847 56203	46579 67540 56308			244 357 309	256 372 313	" " "
	8.88 9.78	25689 33327	19284 33459			141 183	106 184	" "
TOTAL HYDRO-CARBONS (THC)	11.66 13.57	20706 2428	26346 2237			113 13.3	112 12.4	" "

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MINI-1

GC WORKSHEET

COLUMN: 1030 - 1115 ~~1000 - 1100~~ RUN NUMBER: IAF-1 DATE: 8-18-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1	0.29	3512				44.5	0.0127
C_2	0.4	306				3.0	0.0097
C_3	0.66	603				4.2	0.0070
C_4 UK	0.93	293				2.0	as propane
C_5 UK	1.19	860				~~8.1~~ 8.2	"
C_6 CH	1.41	1496				10.5	0.0054
BENZENE UK UK	2.26 3.02	235 4892				1.6 34.2	as propane "
C5 TOLUENE	3.56	3447				14.9	0.0043
XYLENE UK UK	4.02 5.56	1429 780				10.0 5.5	as propane "
UK CH UK	8.61 9.74 11.34	28727 1610 1017	28727			~~200.9~~ 201 60.1 7.1	as propane 0.0037 as propane
	15.31 16.01	226 876				1.6 6.1	" "
TOTAL HYDRO-CARBONS (THC)	16.24 18.17	540 ~~13217~~	13213			4.1 92.4	" "

MIWI-2

GC WORKSHEET

COLUMN: 1030 - 1115 ~~1000-1100~~ RUN NUMBER: IAE-1 DATE: 8-18-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN CTS	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1 UK	.89	7027	6270			38.2 34.0	as benzene 0.0054
C_2 UK	1.03	5126	4364			27.8 23.7	
C_3 C6	1.08	9078	9209			49.3 50.	
C_4 UK	1.19	6407	6159			34.8 33.4	
C_5 Benz	1.35	101626	99804			552 542	
C_6 UK	1.8	25481	24532			138 133	
BENZENE	2.12	164836	162567			895 883	
TOL TOLUENE UK	2.69 3.31	28412 27707	25942 26417			154 141 150 143	
m-xyl o-xyl XYLENE UK	3.87 4.45	133017 50464	134052 48271			657 638 242 230	~~0.0054~~ 0.0018 ~~0.0054~~ 0.0048
	5.17 5.59 6.17	22857 10449 9316	21269 11645 9875			125 116 56.7 63.2 50.6 53.6	as benzene
	7.22 8.05	76091 21355	71784 19005			413 390 116 103	
TOTAL HYDRO-CARBONS (THC)	8.97 10.68 12.72 13.83 14.	44677 29730 3810 15080	41680 28065 3496 15856			243 226 161 152 20.7 13.6 81.9 86.1	

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MIWI-1.

GC WORKSHEET

COLUMN: 13,5 - 14,15 RUN NUMBER: IAF-2 DATE: 8-18-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1	0.39	7477				94.7	as 0.0127
C_2	0.4	420				4.1	0.0090
C_3	0.65	1145				8.0	0.0070
C_4 UK	0.91	425				3.0	as propane 0.0070
C_5 UK	1.17	5397				37.7	"
C_6 C4	1.38	17715				96.5	0.0056
BENZENE UK UK	1.82 2.19	211 235				1.5 1.6	as propane "
TOLUENE UK C5	2.93 3.45	2795.4 16441				19.6 71.0	" 0.0043
XYLENE UK "	3.92 4.65	4349 341				30.4 2.4	0.0070 as propane "
UK UK C6	5.85 8.28 9.28	2241 75707 40006				15.7 529 149	" " 0.0037
UK	13.35 15.55	323 6064				2.3 42.6	as propane "
TOTAL HYDRO-CARBONS (THC)	17.48	15300				107	"

MINI 2

GC WORKSHEET

COLUMN: B15-1415 RUN NUMBER: IAF-2 DATE: 8-18-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1 UIC	1.06	43857				238	ethylene 0.0054
C_2 Ikt	1.16	14992				81.4	
C_3 benz	1.34	203635				1,106	
C_4 UIC	1.77	41172				224	
C_5 TOI	2.06	305939				1661	
C_6 UIC	2.58	42040				200.	0.0048
"	3.18	39090				186	0.0048
BENZENE	3.68	214282				1164	as benzene
	4.22	74977				407	
TOLUENE	4.68	27808				151	
	5.28	14595				79.3	
XYLENE	5.8	10497				57.7	
	6.74	45818				520	
	7.53	24341				132	
	8.36	51091				277	
	9.41	37841				206	
	11.74	7688				41.7	
	12.86	21353				116	
TOTAL HYDRO-CARBONS (THC)							

m-xyl
o-xyl
m-xyl
o-xyl

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MRNZ-1

GC WORKSHEET

COLUMN: 0850-0950 RUN NUMBER: IAE-1 DATE: 8-19-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	~~SPAN~~ CTS	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1	0.29	5043	5323			64.3 67.8	0.0127
C_2	0.4	68	430			6.6 4.1	0.0096
C_3	0.66	1392	1176			8.8 7.4	0.0063
C_4 UK C_4	0.95 1.22	1353 2620	1145 2326			8.6 7.2 16.4 14.7	as ~~benzene~~ propane
C_5 ~~UK~~ ~~C_5~~	1.45 ~~2.35~~ ~~2.35~~ ~~1024~~	6243 ~~6243~~	6028			28.9 27.9	0.0046 as propane
C_6 UK C_6 UK	~~1.63~~ 2.35 ~~2.54~~ 2.63	1024 162	1866 —			6.5 11.8 1. —	" "
BENZENE	~~3.7~~ 3.14	21736	26833			137.4 170.3	" "
TOLUENE	3.7	18952	23661			80.4 100.3	0.0042
XYLENE	~~5.02~~ 4.17 6.32 5.02 753	6499 2839	1587 ~~1601~~ — 1601			41.1 73.3 4.8 18.0 10.1	as propane "
C6	9.02 10.22 12.02	83730 44720 3930	83298 44137 1943			530 527 153 151 24.9 12.3	" 0.0034 as propane
	17.15 19.29	2286 5443	3634 —			14.5 23 34.4	" "
TOTAL HYDRO-CARBONS (THC)							

C4

C5

TRW
ENVIRONMENTAL ENGINEERING DIVISION

M.W. -2

GC WORKSHEET

COLUMN: 0850-0950 RUN NUMBER: IAF-1 DATE: 8-19-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS ~~BENZENE~~ R
C_1 UK	0.88	30313	30185			160 16.0	benzene 0.0057
C_2 UK	1.07	45387	45747			235 242	"
C_3 hex	1.18	18387 ~~16464~~	16948			97.3 89.7	"
C_4 benz	~~1.36~~ 1.34	164631	162561			871 860	"
C_5 UK	1.8	41010	40875			217 216	"
C_6 TOL	2.13	210144	209285			1112 1108	"
BENZENE UK UK	2.67 3.32	35274 31348	34051 31697			187 180 166 168	" "
TOLUENE	M-XYL 3.87 O-XYL 4.45	150718 53474	150344 55183			640 640 223 234	0.0042 0.0042
XYLENE UK	~~4.95~~ 5.17 ~~5.17~~ 5.61	17659 8837	18439 10465			104 103 52.6 214	benzene 0.0053 "
	~~5.61~~ 6.18 ~~6.18~~ 7.2 ~~7.2~~ 8.11	7718 64844 16713	6570 64861 16566			41. 34.8 343 343 88.4 87.7	" " "
	~~8.11~~ 8.69 ~~8.69~~ 10.78	35854 18381	35667 18058			190 189 97.3 95.8	" "
TOTAL HYDRO-CARBONS (THC)	~~10.78~~ 12.78 ~~12.78~~ 14.07 ~~14.07~~	3094 7406	748 2948			11.1 4. 39.2 15.6	!

MIWI-1

GC WORKSHEET

COLUMN: 1030-1130 RUN NUMBER: IAF-2 DATE: 8-19-83

COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE R
C_1	0.29	5623	5806			71.7 74.0	0.0127
C_2	0.41	624	750			6.0 7.6	0.0096
C_3	0.69	1569	1882			5.9 11.9	0.0063
C_4 4B 4K	0.47 1.23	994 3529	1985 4354			6.3 12.6 22.3 27.5	as propane "
C_5 C4	1.47	10246	11679			47.4 54.0	0.0046
C_6 4K 4K2	2.37 3.2	707 26607	553 28209			4.5 3.5 168 178	as propane "
CS BENZENE	3.77	18355	18907			77.7 80.2	0.0042
TOLUENE	4.27 5.13	6497 1342	6168 -			41.1 39 8.5 —	as propane "
XYLENE	5.55 6.48	212 6779	— 1895			1.3 - 42.9 12.0	as propane "
C6 —	9.22 10.42 10.27 12.35	11670 56039 5769	105420 54375 5447			738 667 192.3 187 36.5 21.8	" 0.0034 as propane
	~~9.42~~ 17.42	13.27 14.4 ~~10.43~~ ~~2789~~ 5647	3675 12090			23.2 32.3 76.5	
TOTAL HYDRO-CARBONS (THC)	None						

TRW
ENVIRONMENTAL ENGINEERING DIVISION

MINI-2

GC WORKSHEET

COLUMN: 1030-1130 RUN NUMBER: IAP-2 DATE: 8-19-83

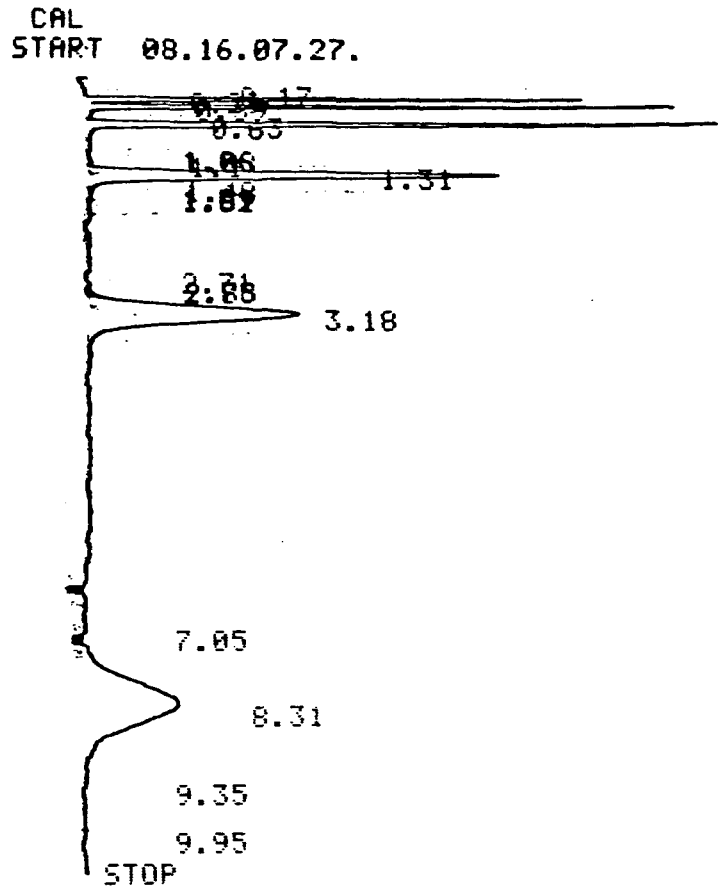
COMPOUND	RETENTION TIME IN CM.	COUNTS	SPAN	ATTENUATION	DILUTION FACTOR (Diluted w/N_2)	CONCENTRATION AS COMPOUND	CONCENTRATION AS BENZENE
C_1 UIL	0.83	34623	34390			183 182	as benzene 0.0053
C_2 UIL	1.03	34694	55801			290 295	"
C_3 Hex	1.2	21931	22023			116 117	"
C_4 Benz	1.38	232785	234264			1232 1240	"
C_5 UIL	1.85	51861	52905			274 280	"
~~UIL~~ TOL	2.18	335213	338609			1774 1797	"
C_6 UIL	α 2.79	41417	41501			222 220	"
~~Spot~~ UIL	~~2.75~~ 3.45	36946	36427			196 195	"
BENZENE	4.03	207555	211332			882 898	0.0042
	4.63	69375	70226			295 298	0.0042
TOLUENE	5.39	21699	22526			115 119	as benzene 0.0053
	5.83	10539	10875			55.8 57.6	"
XYLENE	6.43	3082	8314			42.8 44.2	"
	7.53	72539	72298			384 383	"
	8.41	17070	16760			90.3 88.7	"
	9.37	36337	37633			195 ~~144~~ 199	"
	11.2	18373	18528			97.2 98.1	"
	13.37	571	520			3 2.8	"
TOTAL HYDRO-CARBONS (THC)							

m-xyl

o-xyl

TRW

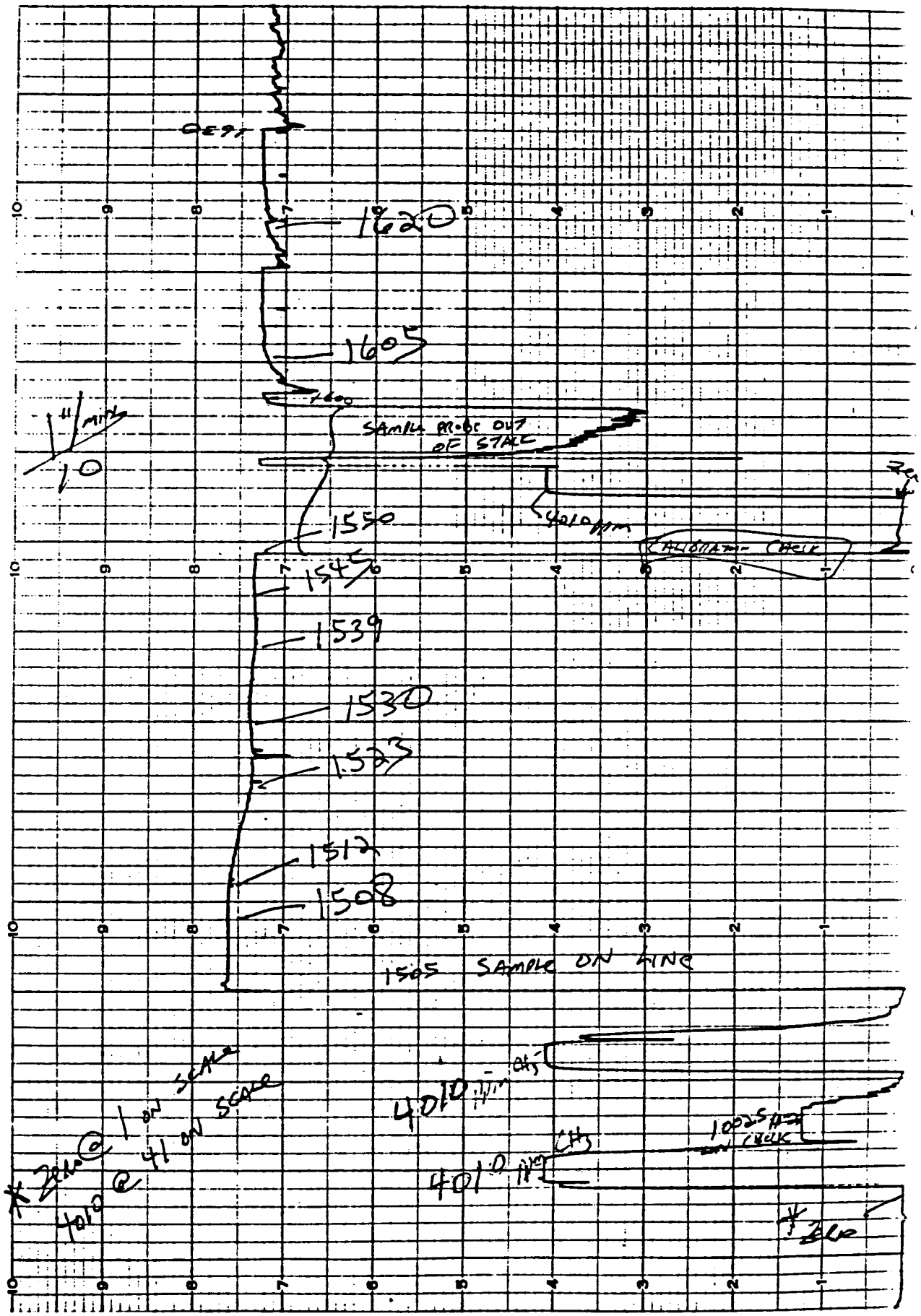
ENVIRONMENTAL ENGINEERING DIVISION



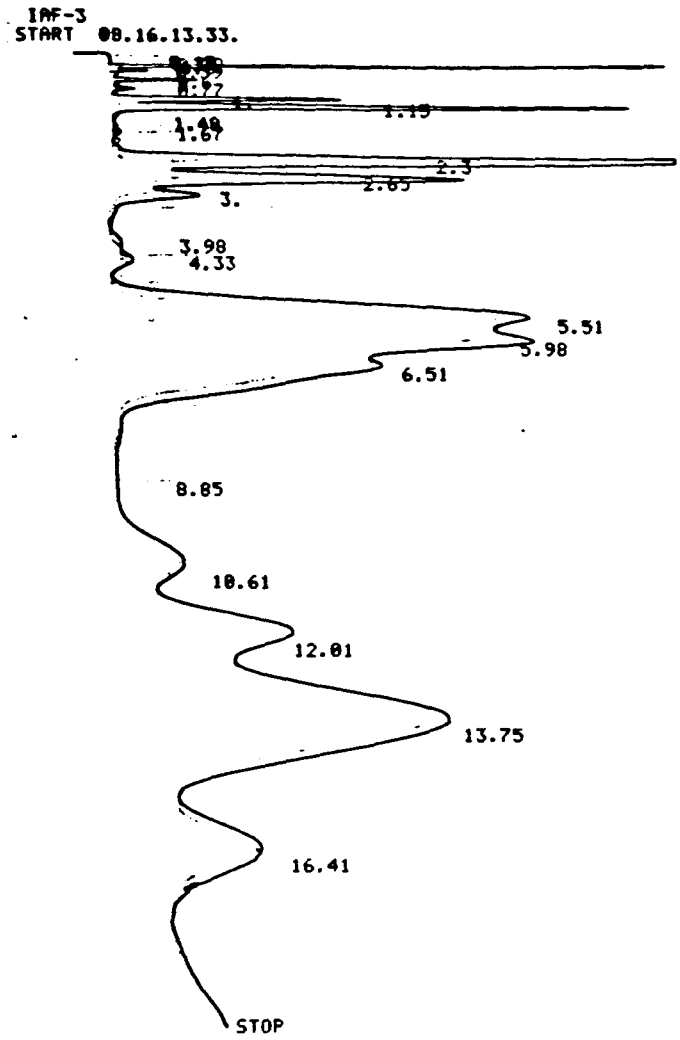
C-R1A
SMPL # 00
FILE # 7
REPT # 118
METHOD 44

#	NAME	TIME	CONC	MK	AREA
		0.17			43
		0.29		V	1167
1	METH	0.39	0	V	1362
2	ETHA	0.63	0	V	1955
		1.31		V	2652
		2.78			27
		3.18		V	3443
		7.05			232
		8.31		V	4556
		9.35		T	25
		9.95		T	94
	TOTAL		0		15560

Example of GC/FID Calibration for C_1-C_6 Speciation



Example of Continuous Monitor Stripchart With a Calibration Sequence:
IAF - Outlet on Test Day 8/15/83

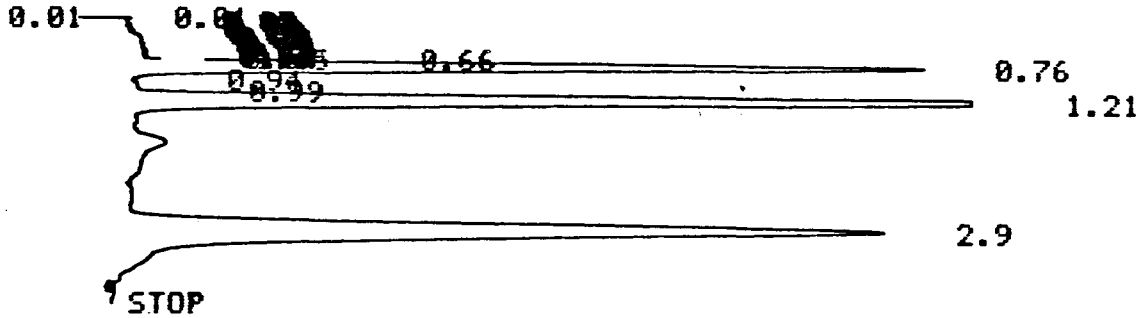


C-R1A
SMPL # 00
FILE # 7
REPT # 127
METHOD 44

#	NAME	TIME	CONC	MK	AREA
		0.13			107
		0.19		V	16
		0.2		V	16
		0.22		V	17
		0.3		V	6734 C1
1	METH	0.39	0	V	931 C2
2	ETHA	0.6	0	V	1887 C3
		0.77		V	913
3	PROP	1.	0	V	7051
		1.15		V	19022 C4
		1.67		T	366
4	BUTA	2.3	0	V	52614
		2.65		V	29131 C5
		3.		V	8099
		3.98			1117
		4.33		V	2987
5	PENT	5.51	0	V	95587
5	PENT	5.98	0	V	91529
		6.51		V	68406 C6
		8.85		T	183
		10.61		V	26906
		12.01		V	68118
		13.75		V	227161
6	HEXA	16.41	0	V	88746
	TOTAL		0		797575

Example of GC/FID Analysis on IAF #3 Gas Bag Sample for C_1-C_6 Speciation

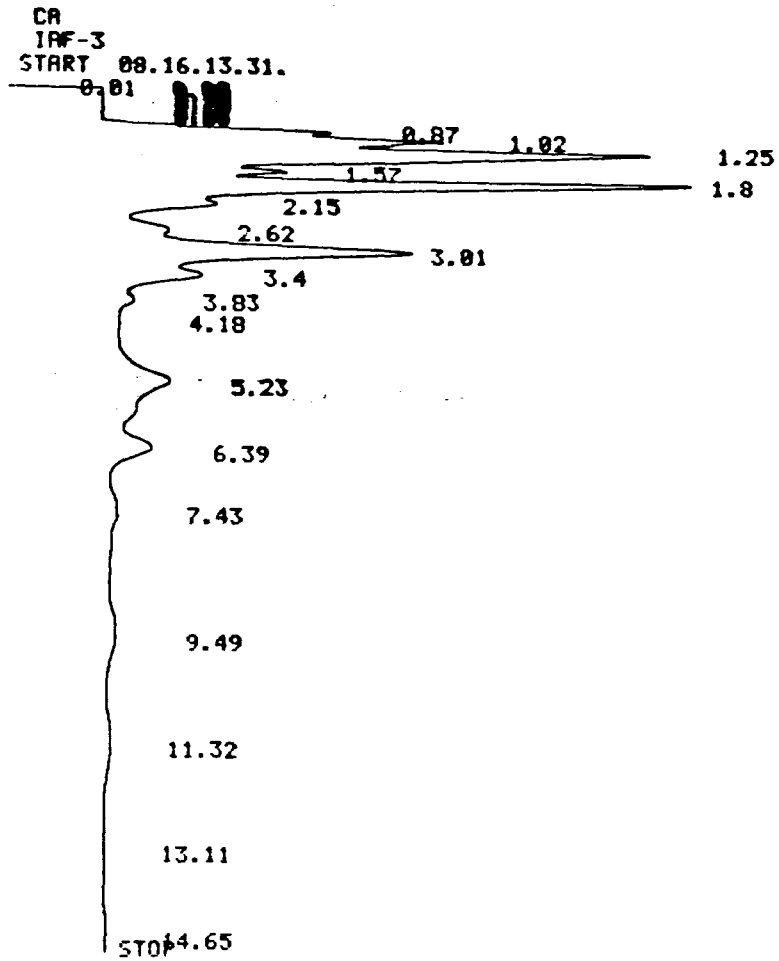
CAL
ATTEN 0
START 08.16.14.23.



C-R1A
SMPL # 00
FILE # 2
REPT # 246
METHOD 44

#	NAME	TIME	CONC	MK	AREA
1	C1-C5	0.76	0	V	5792
		1.21		V	8190
		2.9			8280
	TOTAL		0		22263

Example of GC/FID Calibration for C_6-C_9 Speciation

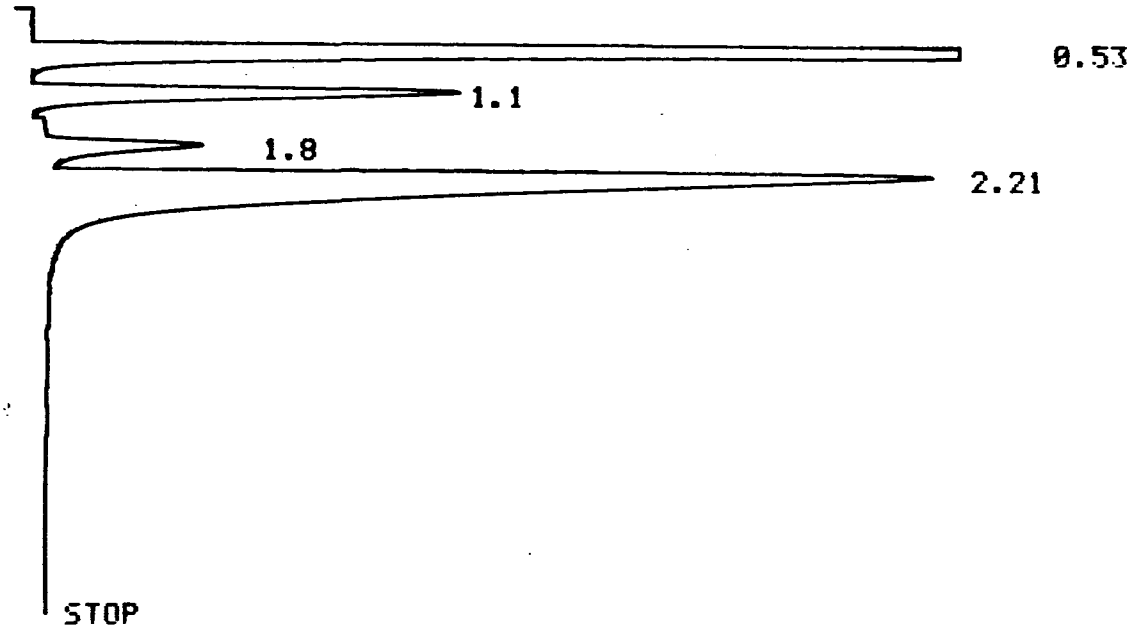


C-R1A
SMPL # 00
FILE # 2
REPT # 243
METHOD 44

#	NAME	TIME	CONC	MK	AREA	
		0.87		V	80538	
		1.02		V	156277	hex
3	BENZ	1.25	0	V	356181	benz
4	HEPT	1.57	0	V	73390	
		1.8		V	341358	tol
5	TOLU	2.15	0	V	66129	
		2.62		V	47801	
		3.01		V	246554	m-xyl
		3.4		V	79865	o-xyl
6	-M XYL	3.83	0	V	26431	
7	ODXYL	4.18	0	V	13640	
		5.23		V	156822	
		6.39		V	63708	
		7.43		V	40513	
		9.49		V	42567	
		11.32		V	23872	
		13.11		V	2408	
		14.65			1079	
	TOTAL		0		1819139	

Example of GC/FID Analysis on IAF #3
Gas Bag Sample for C_6-C_9 Speciation

CAL
START 08.16.14.03.

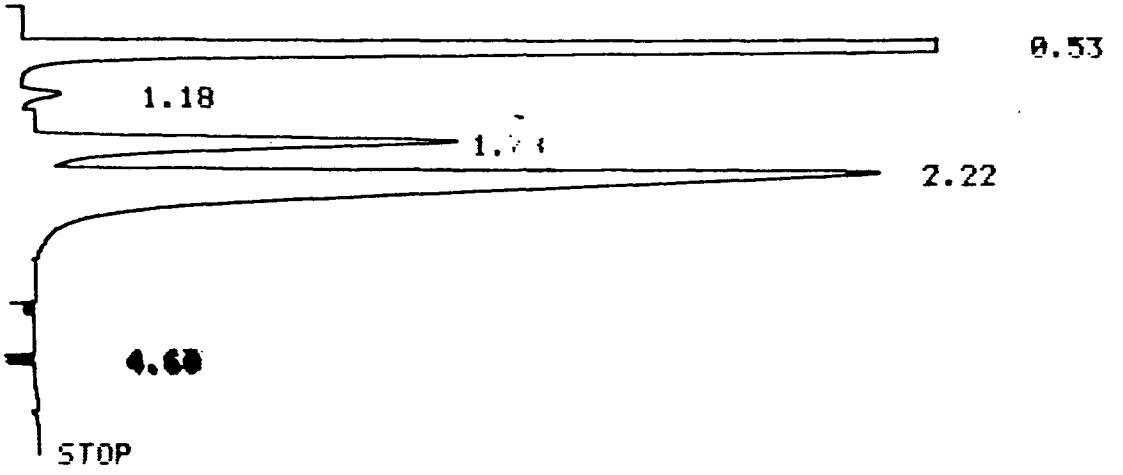


C-R1A
SMPL # 00
FILE # 4
REPT # 1597
METHOD 44

#	NAME	TIME	CONC	MK	AREA
1	AIR	0.53	98.07		47427
2	CP-2	1.1	14.726		8973
3	O-2	1.8	6.0846		3330
4	N-2	2.21	75.4922	V	37134
	TOTAL		194.373		96865

Example of GC/TCD Calibration for Stationary Gas Analysis

IAF-3
START 08.16.13.45.



C-R1A
SMPL # 00
FILE # 4
REPT # 1595
METHOD 44

#	NAME	TIME	CONC	MK	AREA
1	AIR	0.53	110.0757		53233
2	CP-2	1.18	0.9662		588
3	O-2	1.78	18.3754		10058
4	N-2	2.22	72.5525	V	35688
	TOTAL		201.9699		99569

Example of GC/TCD Analysis on IAF #3
Gas Bag Sample for Stationary Gases

APPENDIX D

SAMPLING METHODS AND ANALYTICAL TECHNIQUE

503 OIL AND GREASE

In the determination of oil and grease, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in trichlorotrifluoroethane. "Oil and grease" is any material recovered as a substance soluble in trichlorotrifluoroethane. It includes other material extracted by the solvent from an acidified sample (such as sulfur compounds, certain organic dyes, and chlorophyll) and not volatilized during the test. It is important that this limitation be understood clearly. Unlike some constituents that represent distinct chemical elements, ions, compounds, or groups of compounds, oils and greases are defined by the method used for their determination.

The methods presented here are suitable for biological lipids and mineral hydrocarbons. They also may be suitable for most industrial wastewaters or treated effluents containing these materials, although sample complexity may result in either low or high results because of lack of analytical specificity. The method is not applicable to measurement of low-boiling fractions that volatilize at temperatures below 70 C.

1. Significance

Certain constituents measured by the oil and grease analysis may influence wastewater treatment systems. If present in excessive amounts, they may interfere with aerobic and anaerobic biological processes and lead to decreased wastewater treatment efficiency. When discharged in wastewater or treated effluents, they may cause surface films and shoreline deposits leading to environmental degradation.

A knowledge of the quantity of oil and grease present is helpful in proper design and operation of wastewater treatment systems and also may call attention to certain treatment difficulties.

2. Selection of Method

For liquid samples, three methods are presented: the partition-gravimetric method (A), the partition-infrared method (B), and the Soxhlet method (C). Method B is designed for samples that might contain volatile hydrocarbons that otherwise would be lost in the solvent removal operations of the gravimetric procedure. Method C is the method of choice when relatively polar, heavy petroleum fractions are present, or when the levels of nonvolatile greases may challenge the solubility limit of the solvent. For low levels of oil and grease (<10 mg/L), Method B is the method of choice because gravimetric methods do not provide the needed precision.

Method D is a modification of the Soxhlet Method and is suitable for sludges and similar materials. Method E can be used in

conjunction with Methods A, B, C, or D to obtain a hydrocarbon measurement in addition to, or instead of, the oil and grease measurement. This method separates hydrocarbons from the total oil and grease on the basis of polarity.

3. Sampling and Storage

Collect a representative sample in a wide-mouth glass bottle that has been rinsed with the solvent to remove any detergent film, and acidify in the sample bottle. Collect a separate sample for an oil and grease determination and do not subdivide in the laboratory. When information is required about average grease concentration over an extended period, examine individual portions collected at prescribed time intervals to eliminate losses of grease on sampling equipment during collection of a composite sample.

In sampling sludges, take every possible precaution to obtain a representative sample. When analysis cannot be made immediately, preserve samples with 1 mL conc HCl/80 g sample. Never preserve samples with $CHCl_3$ or sodium benzoate.

503 A. Partition-Gravimetric Method

1. General Discussion

a. Principle: Dissolved or emulsified oil and grease is extracted from water by intimate contact with trichlorotrifluoroethane. Some extractables, especially unsaturated fats and fatty acids, oxidize readily; hence, special precautions regarding temperature and solvent vapor displacement are included to minimize this effect.

b. Interference: Trichlorotrifluoroethane has the ability to dissolve not only oil and grease but also other organic substances. No known solvent will selectively dissolve only oil and grease. Solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatilization. Significant portions of petroleum distillates from gasoline through No. 2 fuel oil are lost in this process. In addition, heavier residuals of petroleum may contain a significant portion of materials that are not extractable with the solvent.

2. Apparatus

a. Separatory funnel, 1 L, with TFE* stopcock.

b. Distilling flask, 125 mL.

c. Water bath.

d. Filter paper, 11 cm diam.†

3. Reagents

a. Hydrochloric acid, HCl, 1 + 1.

b. Trichlorotrifluoroethane‡ (1,1,2-trichloro-1,2,2-trifluoroethane), boiling point 47 C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.

c. Sodium sulfate, Na_2SO_4, anhydrous crystal.

4. Procedure

Collect about 1 L of sample and mark sample level in bottle for later determination of sample volume. Acidify to pH 2 or lower; generally, 5 mL HCl is sufficient. Transfer to a separatory funnel. Carefully rinse sample bottle with 30 mL trichlorotrifluoroethane and add solvent washings to separatory funnel. Preferably shake vigorously for 2 min. However, if it

*Teflon or equivalent.

†Whatman No. 40 or equivalent.

‡Freon or equivalent.

is suspected that a stable emulsion will form, shake gently for 5 to 10 min. Let layers separate. Drain solvent layer through a funnel containing solvent-moistened filter paper into a clean, tared distilling flask. If a clear solvent layer cannot be obtained, add 1 g Na_2SO_4 to the filter paper cone and slowly drain emulsified solvent onto the crystals. Add more Na_2SO_4 if necessary. Extract twice more with 30 mL solvent each but first rinse sample container with each solvent portion. Combine extracts in tared distilling flask and wash filter paper with an additional 10 to 20 mL solvent. Distill solvent from distilling flask in a water bath at 70 C. Place flask on a water bath at 70 C for 15 min and draw air through it with an applied vacuum for the final 1 min. Cool in a desiccator for 30 min and weigh.

5. Calculation

If the organic solvent is free of residue, the gain in weight of the tared distilling flask is mainly due to oil and grease. Total gain in weight, *A*, of tared flask less calculated residue, *B*, from solvent blank is the amount of oil and grease in the sample:

$$\text{mg oil and grease/L} = \frac{(A - B) \times 1{,}000}{\text{mL sample}}$$

6. Precision and Accuracy

Methods A, B, and C were tested by a single laboratory on a sewage sample. By this method the oil and grease concentration was 12.6 mg/L. When 1-L portions of the sewage were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, recovery of added oils was 93% with a standard deviation of 0.9 mg.

503 B. Partition-Infrared Method (TENTATIVE)

1. General Discussion

a. Principle: Although the extraction procedure for this method is identical to that of Method A, infrared detection permits the measurement of many relatively volatile hydrocarbons. Thus, the lighter petroleum distillates, with the exception of gasoline, may be measured accurately. Adequate instrumentation allows for the measurement of as little as 0.2 mg oil and grease/L.

b. Interference: Some degree of selectivity is offered by this method to overcome some of the coextracted interferences discussed in Method A. Heavier residuals of petroleum may contain a significant portion of materials insoluble in trichlorotrifluoroethane.

c. Definitions: A "known oil" is defined as a sample of oil and/or grease that represents the only material of that type used or manufactured in the processes represented by a wastewater. An "unknown oil" is defined as one for which a representative sample of the oil or grease is not available for preparation of a standard.

2. Apparatus

a. Separatory funnel, 1 L, with TFE* stopcock.

b. Infrared spectrophotometer, double beam, recording.

c. Cells, near-infrared silica.

d. Filter paper, 11 cm diam.†

3. Reagents

a. Hydrochloric acid, HCl, 1 + 1.

b. Trichlorotrifluoroethane. See 503A.3b.

c. Sodium sulfate, Na_2SO_4, anhydrous, crystal.

*Teflon or equivalent.

†Whatman No. 40 or equivalent.

d. Reference oil: Prepare a mixture, by volume, of 37.5% iso-octane, 37.5% hexadecane, and 25% benzene. Store in sealed container to prevent evaporation.

4. Procedure

Refer to Method A for sample collection, acidification, and extraction. Collect combined extracts in a 100-mL volumetric flask and adjust final volume to 100 mL with solvent.

Prepare a stock solution of known oil by rapidly transferring about 1 mL (0.5 to 1.0 g) of the oil or grease to a tared 100-mL volumetric flask. Stopper flask and weigh to nearest milligram. Add solvent to dissolve and dilute to mark. If the oil identity is unknown (¶ 1c) use the reference oil (¶ 3d) as the standard. Using volumetric technics, prepare a series of standards over the range of interest. Select a pair of matched near-infrared silica cells. A 1-cm-path-length cell is appropriate for a working range of about 4 to 40 mg. Scan standards and samples from 3,200 cm^{-1} to 2,700 cm^{-1} with solvent in the reference beam and record results on absorbance paper. Measure absorbances of samples and standards by constructing a straight baseline over the scan range and measuring absorbance of the peak maximum at 2,930 cm^{-1} and subtracting baseline absorbance at that point. If the absorbance exceeds 0.8 for a sample, select a shorter pathlength or dilute as required. Use scans of standards to prepare a calibration curve.

5. Calculation

$$\text{mg oil and grease/L} = \frac{A \times 1{,}000}{\text{mL sample}}$$

where:

A = mg of oil or grease in extract as determined from calibration curve.

6. Precision and Accuracy

See 503A.6. By this method the oil and grease concentration was 17.5 mg/L. When 1-L portions of the sewage were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, the recovery of added oils was 99% with a standard deviation of 1.4 mg.

503 C. Soxhlet Extraction Method

1. General Discussion

a. Principle: Soluble metallic soaps are hydrolyzed by acidification. Any oils and solid or viscous grease present are separated from the liquid samples by filtration. After extraction in a Soxhlet apparatus with trichlorotrifluoroethane, the residue remaining after solvent evaporation is weighed to determine the oil and grease content. Compounds volatilized at or below 103 C will be lost when the filter is dried.

b. Interference: The method is entirely empirical and duplicate results can be obtained only by strict adherence to all details. By definition, any material recovered is oil and grease and any filtrable trichlorotrifluoroethane-soluble substances, such as elemental sulfur and certain organic dyes, will be extracted as oil and grease. The rate and time of extraction in the Soxhlet apparatus must be exactly as directed because of varying solubilities of different greases. In addition, the length of time required for drying and cooling extracted material cannot be varied. There may be a gradual increase in weight, presumably due to the absorption of oxygen, and/or a gradual loss of weight due to volatilization.

2. Apparatus

a. Extraction apparatus. Soxhlet.

b. Vacuum pump or other source of vacuum.

c. Buchner funnel, 12 cm.

d. Electric heating mantle.

e. Extraction thimble, paper.

f. Filter paper, 11 cm diam.*

g. Muslin cloth disks, 11 cm diam.

3. Reagents

a. Hydrochloric acid. HCl, 1 + 1.

b. Trichlorotrifluoroethane: See 503A.3*b*.

c. Diatomaceous-silica filter aid suspension.† 10 g/L distilled water.

4. Procedure

Collect about 1 L of sample in a wide-mouth glass bottle and mark sample level in bottle for later determination of sample volume. Acidify to pH 2 or lower; generally, 5 mL HCl is sufficient. Prepare a filter consisting of a muslin cloth disk overlaid with filter paper. Wet paper and muslin and press down edges of paper. Using a vacuum, pass 100 mL filter aid suspension through prepared filter and wash with 1 L distilled water. Apply vacuum until no more water passes filter. Filter acidified sample. Apply vacuum until no more water passes through filter. Using forceps, transfer filter paper to a watch glass. Add material adhering to edges of muslin cloth disk. Wipe sides and bottom of collecting vessel and Buchner funnel with pieces of filter paper soaked in solvent, taking care to remove all films caused by grease and to collect all solid material. Add pieces of filter paper to filter paper on watch glass. Roll all filter paper containing sample and fit into a paper extraction thimble. Add any pieces of material remaining on watch glass. Wipe watch glass with a filter paper soaked in solvent and place in paper extraction thimble. Dry filled thimble in a hot-air oven at 103 C for 30 min. Fill thimble with glass wool or small glass beads. Weigh extraction flask. Extract oil and grease in a Soxhlet apparatus, using trichlorotrifluoroethane at a rate of 20 cycles/hr for 4 hr. Time from first cycle. Distill solvent from extraction flask in a water bath at 70 C. Place flask on a water bath at 70 C for 15 min and draw air through it using an applied vacuum for the final 1 min. Cool in a desiccator for 30 min and weigh.

5. Calculation

See Section 503A.5.

6. Precision and Accuracy

See Section 503A.6. By this method the oil and grease concentration was 14.8 mg/L. When 1-L portions of the sewage were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, the recovery of added oils was 88% with a standard deviation of 1.1 mg.

*Whatman No. 40 or equivalent.

†Hyflo Super-Cel, Johns-Manville Corp., or equivalent.

503 D. Extraction Method for Sludge Samples

1. General Discussion

a. Principle: Drying acidified sludge by heating leads to low results. Magnesium sulfate monohydrate is capable of combining with 75% of its own weight in water in forming $MgSO_4 \cdot 7H_2O$ and is used to dry sludge. After drying, the oil and grease can be extracted with trichlorotrifluoroethane.

b. Interference: See 503C.1*b*.

2. Apparatus

a. Extraction apparatus. Soxhlet.

b. Vacuum pump or other source of vacuum.

c. Extraction thimble. paper.

d. Grease-free cotton: Extract non-absorbent cotton with solvent.

3. Reagents

a. Hydrochloric acid. HCl, conc.

b. Magnesium sulfate monohydrate: Prepare $MgSO_4 \cdot H_2O$ by overnight drying of a thin layer in an oven at 150 C.

c. Trichlorotrifluoroethane: See 503A.3*b*.

4. Procedure

In a 150-mL beaker weigh a sample of wet sludge, 20 ± 0.5 g, of which the dry-solids content is known. Acidify to pH 2.0 (generally, 0.3 mL conc HCl is sufficient). Add 25 g $MgSO_4 \cdot H_2O$. Stir to a smooth paste and spread on sides of beaker to facilitate subsequent removal. Let stand until solidified, 15 to 30 min. Remove solids and grind in a porcelain mortar. Add the powder to a paper extraction thimble. Wipe beaker and mortar with small pieces of filter paper moistened with solvent and add to thimble. Fill thimble with glass wool or small glass beads. Extract in a Soxhlet apparatus, using trichlorotrifluoroethane, at a rate of 20 cycles/hr for 4 hr. If any turbidity or suspended matter is present in the extraction flask, remove by filtering through grease-free cotton into another weighed flask. Rinse flask and cotton with solvent. Distill solvent from extraction flask in water at 70 C. Place flask on a water bath at 70 C for 15 min and draw air through it using an applied vacuum for the final 1 min. Cool in a desiccator for 30 min and weigh.

5. Calculation

$$\text{Oil and grease as \% of dry solids} = \frac{\text{gain in weight of flask, g} \times 100}{\text{weight of wet solids, g} \times \text{dry solids fraction}}$$

6. Precision

The examination of six replicate samples of sludge yielded a standard deviation of 4.6%.

503 E. Hydrocarbons

1. Significance

In the absence of specially modified industrial products, oil and grease is composed primarily of fatty matter from animal and vegetable sources and hydrocarbons of petroleum origin. A knowledge of the percentage of each of these constituents in the total oil and grease minimizes the difficulty in determining the major source of the material and simplifies the correction of oil and grease problems in wastewater treatment plant operation and stream pollution abatement.

2. General Discussion

a. Principle: Silica gel has the ability to absorb polar materials. If a solution of hydrocarbons and fatty materials in trichlorotrifluoroethane is mixed with silica gel, the fatty acids are selectively removed from solution. The materials not eliminated by silica gel adsorption are designated hydrocarbons by this test.

b. Interference: The more polar hydrocarbons, such as complex aromatic compounds and hydrocarbon derivatives of chlorine, sulfur, and nitrogen, may be ad-

rection is unnecessary if dilution water meets the blank criteria stipulated above. If the dilution water does not meet these criteria, proper corrections are difficult and results become questionable.

7. Precision and Accuracy

In a series of interlaboratory studies, each involving 86 to 102 laboratories (and as many river water and wastewater seeds), 5-day BOD measurements were made on synthetic water samples containing a 1:1 mixture of glucose and glutamic acid in the total concentration range of 5 to 340 mg/L. The regression equations for mean value, $\bar{X}$, and standard deviation, S, from these studies were:

$$\bar{X} = 0.665\,(\text{added level, mg/L}) - 0.149$$

$$S = 0.120\,(\text{added level, mg/L}) + 1.04$$

For the 300-mg/L mixed primary standard, the average 5-day BOD was 199.4 mg/L with a standard deviation of 37.0 mg/L.

8. References

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508 OXYGEN DEMAND (CHEMICAL)

The chemical oxygen demand (COD) is a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific source, COD can be related empirically to BOD, organic carbon, or organic matter content.

1. Selection of Method

The dichromate reflux method is preferred over other methods using oxidants because of superior oxidizability, applicability to a wide variety of samples, and ease of manipulation. The test is most useful for monitoring and control, especially after correlations with constituents[1,2] such as BOD and organic carbon have been developed. For most organic compounds oxidation is 95 to 100% of the theoretical value.[2,3] Pyridine is not oxidized.[2] Benzene and other volatile organics are oxi-

dized if they have sufficient contact with the oxidants.[2] While the carbonaceous portion of nitrogen-containing organic matter is oxidized, no oxidation of ammonia, either present in a waste or liberated from the nitrogen-containing organic matter, takes place in the absence of significant chloride concentrations.

2. Sampling and Storage

Test unstable samples without delay. Homogenize samples containing settleable solids in a blender to permit representative sampling. If there is to be a delay before analysis, preserve the sample by acidification to pH 2 or lower with conc sulfuric acid (H_2SO_4). Make preliminary dilutions for wastes containing a high COD to reduce the error inherent in measuring small volumes of sample.

508 A. Dichromate Reflux Method

1 General Discussion

a. Principle: Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion the remaining unreduced $K_2Cr_2O_7$ is titrated with ferrous ammonium sulfate (FAS), the amount of $K_2Cr_2O_7$ consumed is determined, and the amount of oxidizable organic matter is calculated in terms of oxygen equivalent.

b. Interferences and limitations: Volatile straight-chain aliphatic compounds are not oxidized to any appreciable extent. This failure occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight-chain aliphatic compounds are oxidized more effectively when silver sulfate (Ag_2SO_4) is added as a catalyst. However, Ag_2SO_4 reacts with chloride, bromide, and iodide to produce precipitates that are oxidized only partially. The difficulties caused by the presence of halides can be largely, though not completely, overcome by complexing with mercuric sulfate ($HgSO_4$) before the refluxing procedure.[1] Do not use the test for samples containing more than 2,000 mg chloride/L.

Nitrite (NO_2^-) exerts a COD of 1.1 mg O_2/mg NO_2-N. Because concentrations of NO_2^- in polluted waters rarely exceed 1 or 2 mg NO_2-N/L the interference is considered insignificant and usually is ignored. To eliminate a significant interference due to NO_2^-, add 10 mg sulfamic acid/mg NO_2-N present in the refluxing flask. Also add the same amount of sulfamic acid to the reflux flask containing the distilled water blank.

Reduced inorganic species such as ferrous iron, sulfide, manganous manganese, etc., are oxidized quantitatively under the test conditions. For samples containing significant levels of these species, stoichiometric oxidation can be assumed from known initial concentration of the interfering species and corrections can be made to the COD value obtained.

c. Minimum detectable concentration: Determine COD values of >50 mg/L using 0.250N $K_2Cr_2O_7$. With 0.025N $K_2Cr_2O_7$, COD values from 5 to 50 mg/L can be determined but with lesser accuracy.[3]

2. Apparatus

Reflux apparatus, consisting of 500-mL

or 250-mL erlenmeyer flasks with ground-glass 24/40 necks* and 300-mm jacket Liebig, West, or equivalent condensers,† with 24/40 ground-glass joint, and a hot plate having sufficient power to produce at least 1.4 W/cm² of heating surface, or equivalent.

3. Reagents

a. Standard potassium dichromate solution, 0.250N: Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 103 C for 2 hr, in distilled water and dilute to 1,000 mL.

b. Silver sulfate, Ag_2SO_4, reagent or technical grade, crystals or powder.

c. Sulfuric acid reagent: Add Ag_2SO_4 to conc H_2SO_4 at the rate of 22 g Ag_2SO_4/4 kg bottle. Let stand 1 to 2 days to dissolve Ag_2SO_4.

d. Sulfuric acid, H_2SO_4, conc.

e. Ferroin indicator solution: Dissolve 1.485 g 1,10-phenanthroline monohydrate and 695 mg $FeSO_4 \cdot 7H_2O$ in distilled water and dilute to 100 mL. This indicator solution may be purchased already prepared.‡

f. Standard ferrous ammonium sulfate titrant, approximately 0.25N: Dissolve 98 g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (FAS) in distilled water. Add 20 mL conc H_2SO_4, cool, and dilute to 1,000 mL. Standardize this solution daily against standard $K_2Cr_2O_7$ solution, as follows:

Dilute 10.0 mL standard $K_2Cr_2O_7$ solution to about 100 mL. Add 30 mL conc H_2SO_4 and cool. Titrate with FAS titrant, using 0.10 to 0.15 mL (2 to 3 drops) ferroin indicator.

$$\text{Normality of FAS solution} = \frac{\text{Volume 0.25N } K_2Cr_2O_7 \text{ solution titrated, mL}}{\text{Volume FAS used in titration, mL}} \times 0.25$$

g. Mercuric sulfate: $HgSO_4$, crystals or powder.

h. Sulfamic acid: Required only if the interference of nitrites is to be eliminated (see ¶ 1*b* above).

i. Potassium hydrogen phthalate standard: Lightly crush and then dry potassium acid phthalate ($HOOCC_6H_4COOK$) to constant weight at 120 C, dissolve 425 mg in distilled water, and dilute to 1,000 mL. Potassium hydrogen phthalate has a theoretical COD of 1.176 g O_2/g and this solution has a theoretical COD of 500 mg O_2/L. Prepare fresh for each use.

4. Procedure

a. Treatment of samples with ≤50 mg COD/L: Place 50.0 mL sample (for samples with COD >900 mg COD/L, use a smaller sample portion diluted to 50.0 mL) in the 500-mL refluxing flask. Add 1 g $HgSO_4$, several glass beads, and very slowly add 5.0 mL sulfuric acid reagent, with mixing to dissolve $HgSO_4$. Cool while mixing to avoid possible loss of volatile materials. Add 25.0 mL 0.250N $K_2Cr_2O_7$ solution and mix. Attach flask to condenser and turn on cooling water. Add remaining sulfuric acid reagent (70 mL) through open end of condenser. Continue swirling and mixing while adding sulfuric acid reagent. CAUTION: *Mix reflux mixture thoroughly before applying heat to prevent local heating of flask bottom and a possible blowout of flask contents.* If sample volumes other than 50 mL are used, keep ratios of reagent weights, volumes, and strengths constant. See Table 508:I for examples of applicable ratios. Maintain these ratios and follow the procedure as outlined above.

Use 1 g $HgSO_4$ with a 50.0-mL sample to complex up to a maximum of 100 mg chloride (2,000 mg/L). For smaller samples use less $HgSO_4$, according to the chloride concentration; maintain a 10:1 ratio of $HgSO_4$:Cl. A slight precipitate does not affect the determination adversely. Gener-

*Corning 5000 or equivalent.

†Corning 2360, 91548, or equivalent.

‡G. F. Smith Chemical Co., Columbus, Ohio.

TABLE 508:1. REAGENT QUANTITIES AND NORMALITIES FOR VARIOUS SAMPLE SIZES

Sample Size *mL*	0.25N Standard Dichromate *mL*	Sulfuric Acid Reagent *mL*	$HgSO_4$ *g*	Normality of FAS	Final Volume before Titration *mL*
10.0	5.0	15	0.2	0.05	70
20.0	10.0	30	0.4	0.10	140
30.0	15.0	45	0.6	0.15	210
40.0	20.0	60	0.8	0.20	280
50.0	25.0	75	1.0	0.25	350

ally. COD cannot be measured accurately in samples containing more than 2,000 mg chloride/L.

Reflux mixture for 2 hr. Use a shorter period for particular wastes if it has been shown that the shorter period yields the same COD as that found by 2-hr refluxing. Cover open end of condenser with a small beaker to prevent foreign material from entering refluxing mixture. Cool and wash down condenser with distilled water.

Disconnect reflux condenser and dilute mixture to about twice its volume with distilled water. Cool to room temperature and titrate excess $K_2Cr_2O_7$ with FAS, using 0.10 to 0.15 mL (2 to 3 drops) ferroin indicator. Although the quantity of ferroin indicator is not critical, use the same volume for all titrations. Take as the end point of the titration the first sharp color change from blue-green to reddish brown. The blue-green may reappear.

Reflux and titrate in the same manner a blank containing the reagents and a volume of distilled water equal to that of sample.

b. Alternate procedure for low-COD samples: Follow the above procedure, ¶ 4*a*, with two exceptions: (*i*) Use standard 0.025N $K_2Cr_2O_7$, and (*ii*) titrate with 0.025N FAS. Exercise extreme care with this procedure because even a trace of organic matter on glassware or from the atmosphere may cause gross errors.

If a further increase in sensitivity is required, concentrate a larger volume of sample before digesting under reflux as follows: Add all reagents to a sample larger than 50 mL and reduce total volume to 150 mL by boiling in the refluxing flask open to the atmosphere without the condenser attached. Compute amount of $HgSO_4$ to be added (before concentration) on the basis of a weight ratio of 10:1, $HgSO_4$:Cl, using the amount of chloride present in the original volume of sample. Carry a blank reagent through the same procedure.

This technic has the advantage of concentrating the sample without significant losses of easily digested volatile materials. Hard-to-digest volatile materials such as volatile acids are lost, but an improvement is gained over ordinary evaporative concentration methods.

c. Determination of standard solution: Evaluate the technic and quality of reagents by testing a standard potassium hydrogen phthalate solution.

5. Calculation

$$\text{mg COD/L} = \frac{(A - B) \times N \times 8{,}000}{\text{mL sample}}$$

where:

A = volume FAS used for blank, mL,
B = volume FAS used for sample, mL, and
N = normality of FAS.

6. Precision and Accuracy

A set of synthetic samples containing potassium hydrogen phthalate and NaCl was tested by 74 laboratories.[3] At 200 mg COD/L in the absence of chloride, the standard deviation was ± 13 mg/L (coefficient of variation, 6.5%). At 160 mg COD/L and 100 mg chloride/L the standard deviation was ± 14 mg/L (coefficient of variation, 10.8%).

508 B. References

1. MOORE, W.A., R. C. KRONER & C.C. RUCHHOFT. 1949. Dichromate reflux method for determination of oxygen consumed. *Anal. Chem.* 21:953.
2. MOORE, W.A., F. J. LUDZACK & C.C. RUCHHOFT. 1951. Determination of oxygen-consumed values of organic wastes. *Anal. Chem.* 23:1297.
3. MEDALIA, A.I. 1951. Test for traces of organic matter in water. *Anal. Chem.* 23:1318.
4. DOBBS, R.A. & R.T. WILLIAMS. 1963. Elimination of chloride interference in the chemical oxygen demand test. *Anal. Chem* 35:1064.
5. ANALYTICAL REFERENCE SERVICE, USHEW-PHS. 1965. Oxygen Demand No. 2. Study No. 21, Environmental Health Ser., Water. PHS Publ. No. 999-WP-26.

ORGANIC CARBON, TOTAL

Method 415.1 (Combustion or Oxidation)

STORET NO. Total 00680
Dissolved 00681

1. Scope and Application
   1.1 This method includes the measurement of organic carbon in drinking, surface and saline waters, domestic and industrial wastes. Exclusions are noted under Definitions and Interferences.
   1.2 The method is most applicable to measurement of organic carbon above 1 mg/l.
2. Summary of Method
   2.1 Organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion or wet chemical oxidation. The CO_2 formed can be measured directly by an infrared detector or converted to methane (CH_4) and measured by a flame ionization detector. The amount of CO_2 or CH_4 is directly proportional to the concentration of carbonaceous material in the sample.
3. Definitions
   3.1 The carbonaceous analyzer measures all of the carbon in a sample. Because of various properties of carbon-containing compounds in liquid samples, preliminary treatment of the sample prior to analysis dictates the definition of the carbon as it is measured. Forms of carbon that are measured by the method are:
      A) soluble, nonvolatile organic carbon; for instance, natural sugars.
      B) soluble, volatile organic carbon; for instance, mercaptans.
      C) insoluble, partially volatile carbon; for instance, oils.
      D) insoluble, particulate carbonaceous materials, for instance; cellulose fibers.
      E) soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter; for instance, oily matter adsorbed on silt particles.
   3.2 The final usefulness of the carbon measurement is in assessing the potential oxygen-demanding load of organic material on a receiving stream. This statement applies whether the carbon measurement is made on a sewage plant effluent, industrial waste, or on water taken directly from the stream. In this light, carbonate and bicarbonate carbon are not a part of the oxygen demand in the stream and therefore should be discounted in the final calculation or removed prior to analysis. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon which are measured. Instrument manufacturer's instructions should be followed.

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4. Sample Handling and Preservation

4.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.
NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

4.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

4.3 In instances where analysis cannot be performed within two hours (2 hours) from time of sampling, the sample is acidified (pH ≤ 2) with HCl or H_2SO_4.

5. Interferences

5.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.

5.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter type syringe or pipette. The openings of the syringe or pipette limit the maximum size of particles which may be included in the sample.

6. Apparatus

6.1 Apparatus for blending or homogenizing samples: Generally, a Waring-type blender is satisfactory.

6.2 Apparatus for total and dissolved organic carbon:

6.2.1 A number of companies manufacture systems for measuring carbonaceous material in liquid samples. Considerations should be made as to the types of samples to be analyzed, the expected concentration range, and forms of carbon to be measured.

6.2.2 No specific analyzer is recommended as superior.

7. Reagents

7.1 Distilled water used in preparation of standards and for dilution of samples should be ultra pure to reduce the carbon concentration of the blank. Carbon dioxide-free, double distilled water is recommended. Ion exchanged waters are not recommended because of the possibilities of contamination with organic materials from the resins.

7.2 Potassium hydrogen phthalate, stock solution, 1000 mg carbon/liter: Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 ml.
NOTE 2: Sodium oxalate and acetic acid are not recommended as stock solutions.

7.3 Potassium hydrogen phthalate, standard solutions: Prepare standard solutions from the stock solution by dilution with distilled water.

7.4 Carbonate-bicarbonate, stock solution, 1000 mg carbon/liter: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100 ml volumetric flask. Dissolve with distilled water.

7.5 Carbonate-bicarbonate, standard solution: Prepare a series of standards similar to step 7.3.

NOTE 3: This standard is not required by some instruments.

7.6 Blank solution: Use the same distilled water (or similar quality water) used for the preparation of the standard solutions.

8. Procedure

8.1 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

8.2 For calibration of the instrument, it is recommended that a series of standards encompassing the expected concentration range of the samples be used.

9. Precision and Accuracy

9.1 Twenty-eight analysts in twenty-one laboratories analyzed distilled water solutions containing exact increments of oxidizable organic compounds, with the following results:

Increment as TOC mg/liter	Precision as Standard Deviation TOC, mg/liter	Accuracy as Bias, %	Accuracy as Bias, mg/liter
4.9	3.93	+15.27	+0.75
107	8.32	+ 1.01	+1.08

(FWPCA Method Study 3, Demand Analyses)

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D 2574–79, p 469 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 532, Method 505, (1975).

APPENDIX E

TEST LOG

APPENDIX E

TEST LOG

Date	Time	Task Performed
8/15/83	0900	TRW test crew and EPA representative arrive at the Golden West Refinery facility in Santa Fe Springs, California.
	0930	Crew begins set-up at test sites. Some problem with electricity supply.
	1300	Continuous hydrocarbon monitor (Beckman 402) placed on-line for testing at the IAF-outlet sample location.
	1400	Problem with electricity supply.
	1600	Beckman 402 back on-line at IAF-outlet.
	1730	Crew departs test facility.
8/16/83	0700	TRW test crew and EPA representative arrive at the Golden West facility.
	0735	IAF gas bag sample #1.
	0805	Liquid VOA sample taken and composite sample began at IAF-inlet.
	0810	Liquid VOA sample taken and composite began at IAF-outlet.
	0830	Liquid composite sample at API-inlet.
	1020	IAF gas bag sample #2.
	1235	IAF gas bag sample #3.
	1400	Liquid VOA samples at IAF-inlet and IAF-outlet.
	1600	Crew departs test facility.
8/17/83	0700	TRW test crew and EPA representative arrive at the Golden West facility.
	0740	Liquid VOA and composite samples at IAF-inlet, IAF-outlet, and API-inlet (composite only).
	0745	IAF gas bag sample #1.
	0830	Perform monitoring check of drain system with EPA and NSPS representatives.

APPENDIX E (Concluded)

Date	Time	Task Performed
8/17/83	1000	IAF gas bag sample #2.
	1153	IAF gas bag sample #3.
	1300	Liquid VOA samples at IAF-inlet and IAF-outlet.
	1600	Crew departs test facility.
8/18/83	0700	TRW test crew and EPA representative arrive at the Golden West facility.
	0800	Process interruption - instruments off-line.
	1000	Process stable - instrument back on-line.
	1030	IAF gas bag sample #1.
	1040	Liquid VOA and composite samples at IAF-inlet and IAF-outlet.
	1130	Liquid composite sample at API-inlet.
	1310	IAF gas bag sample #2.
	1500	Crew departs test facility.
8/19/83	0730	TRW test crew and EPA representative arrive at the Golden West facility.
	0800	Perform monitoring check of drain system with EPA and NSPS representative.
	0830	Liquid VOA and composite samples at the IAF-inlet, IAF-outlet and API-inlet (composite only).
	0850	IAF gas bag sample #1.
	1030	IAF gas bag sample #2.
	1400	Liquid VOA samples at the IAF-inlet and IAF-outlet.
		Instruments off-line and flame-out.
		End of test period.
	1530	Crew departs test facility.

APPENDIX F

PROJECT PARTICIPANTS

APPENDIX F

PROJECT PARTICIPANTS

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